STIC SEARCH RESULTS FEEDBACK FORM

USPTO

Biotech-Chem Library

Questions about the scope or the results of the search? Contact ***the searcher or contact*:**

Mary Hale, Information Branch Supervisor
Remsen Bldg. 01 D86
571-272-2507

Voluntary Results Feedback Form

- *I am an examiner in Workgroup:* ☐ *Example: 1610*
- *Relevant prior art* ***found****, search results used as follows:*
  - ☐ 102 rejection
  - ☐ 103 rejection
  - ☐ Cited as being of interest.
  - ☐ Helped examiner better understand the invention.
  - ☐ Helped examiner better understand the state of the art in their technology.

  Types of relevant prior art found:
  - ☐ Foreign Patent(s)
  - ☐ Non-Patent Literature
    (journal articles, conference proceedings, new product announcements etc.)
- *Relevant prior art* ***not found:***
  - ☐ Results verified the lack of relevant prior art (helped determine patentability).
  - ☐ Results were not useful in determining patentability or understanding the invention.

Comments:

Drop off or send completed forms to STIC-Biotech-Chem Library Remsen Bldg.

sira
Search and Information Resources Administration

THIS PAGE IS BLANK

ACCESS DB # 185030 5.3.06

FOR OFFICIAL USE ONLY

PLEASE PRINT CLEARLY

Scientific and Technical Information Center

SEARCH REQUEST FORM

Requester's Full Name: Rei-Tzu (Robert) Shiao Examiner # : 79521 Date: 4/12/06
Art Unit: 1626 Phone Number: 2-0707 Serial Number: 10/734,208
Location (Bldg/Room#): REM (Mailbox #): 5A10/5C18 Results Format Preferred (circle): PAPER DISK

****

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: Synthesis of mono-N-substituted

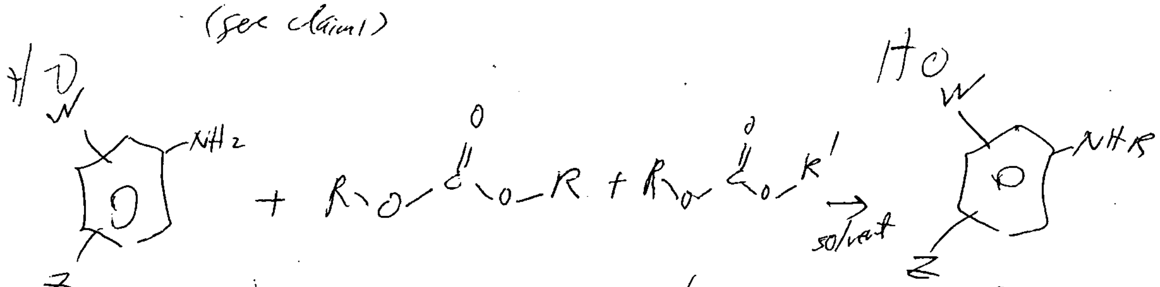
Inventors (please provide full names): Selva et al

Earliest Priority Date: ______________________

Search Topic:
Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

For Sequence Searches Only *Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.*

I. step in process making cpd I. by
(see claim 1)



(II) (III) (IV) (I)

* W is OH, $-CH_2OH$, $-COOH$, $-CONH_2$
* Z is H, halogen, alkyl, alkoxy, NO_2, $-CN$
* R is alkylene
* R' is $CH_3(OCH_2CH_2)_n$, n ≥ 2 or alkyl

****

STAFF USE ONLY

Searcher: ______________________
Searcher Phone #: ______________________
Searcher Location: ______________________
Date Searcher Picked Up: ______________________
Date Completed: ______________________
Searcher Prep & Review Time: ______________________
Online Time: ______________________

Type of Search

_____ NA Sequence (#)
_____ AA Sequence (#)
_____ Structure (#)
_____ Bibliographic
_____ Litigation
_____ Fulltext
_____ Other

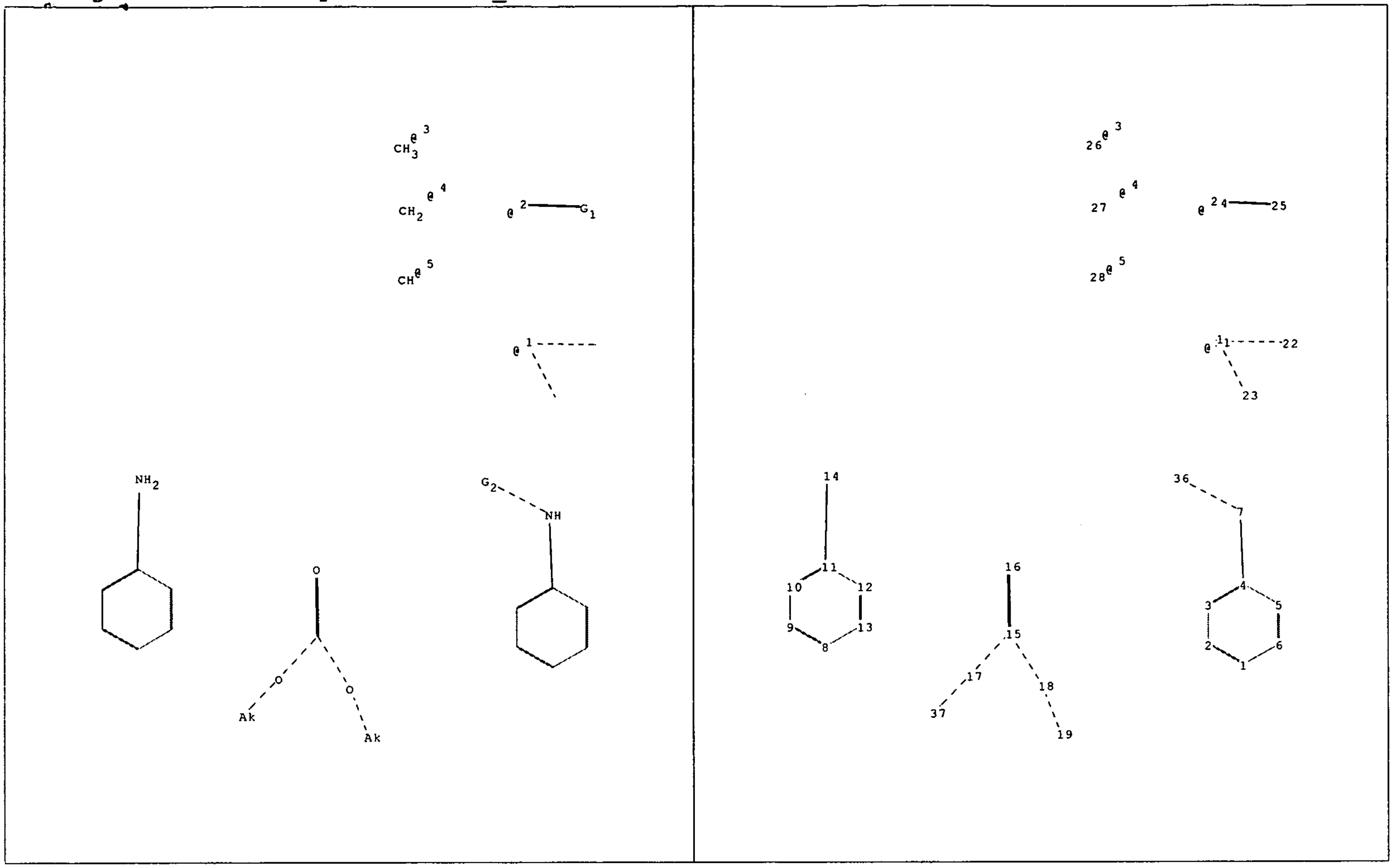
Vendors and cost where applicable

_____ STN _____ Dialog
_____ Questel/Orbit _____ Lexis/Nexis
_____ Westlaw _____ WWW/Internet
_____ In-house sequence systems
___ Commercial ___ Oligomer ___ Score/Length
___ Interference ___ SPDI ___ Encode/Transl
_________ Other (specify)

THIS PAGE IS BLANK

C:\Program Files\Stnexp\Queries\10_734208\shiao-brx.str

STRUCTURE "L5"



chain nodes :
7 14 15 16 17 18 19 21 22 23 24 25 26 27 28 36 37
ring nodes :
1 2 3 4 5 6 8 9 10 11 12 13
chain bonds :
4-7 7-36 11-14 15-16 15-17 15-18 17-37 18-19 21-22 21-23 24-25
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 8-9 8-13 9-10 10-11 11-12 12-13
exact/norm bonds :
4-7 7-36 11-14 15-16 15-17 15-18 17-37 18-19 21-22 21-23 24-25
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6 8-9 8-13 9-10 10-11 11-12 12-13
isolated ring systems :
containing 1 : 8 :

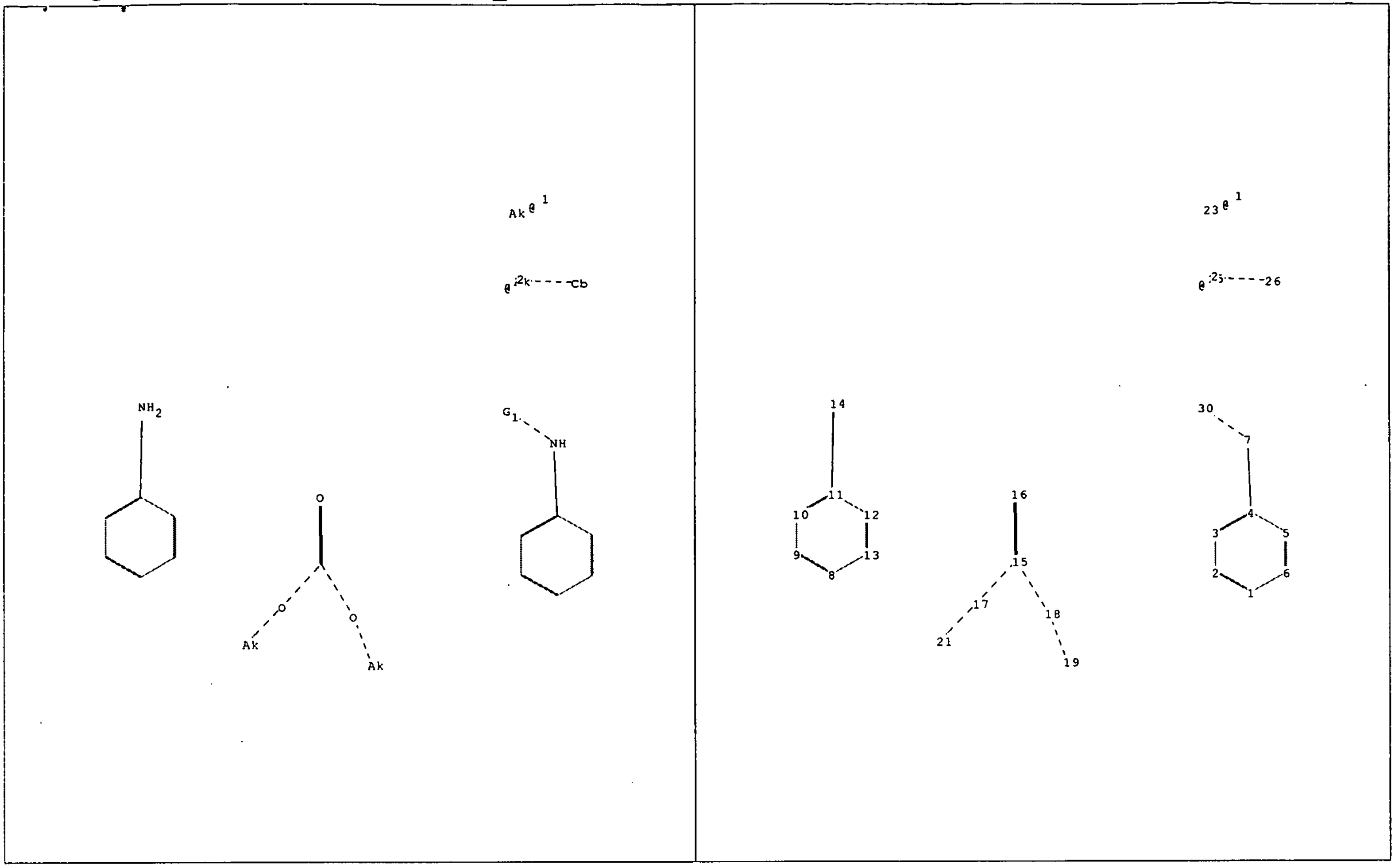
G1:C,N

G2:[*1],[*2],[*3],[*4],[*5]

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:Atom 9:Atom 10:Atom 11:Atom
12:Atom 13:Atom 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS 19:CLASS 21:CLASS
22:CLASS 23:CLASS 24:CLASS 25:CLASS 26:CLASS 27:CLASS 28:CLASS 36:CLASS 37:CLASS
fragments assigned product role:
containing 1
fragments assigned reactant/reagent role:

containing 8
containing 15

C:\Program Files\Stnexp\Queries\10_734208\shiao-crx.str STRUCTURE "L34"



chain nodes :
7 14 15 16 17 18 19 21 23 25 26 30
ring nodes :
1 2 3 4 5 6 8 9 10 11 12 13
chain bonds :
4-7 7-30 11-14 15-16 15-17 15-18 17-21 18-19 25-26
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 8-9 8-13 9-10 10-11 11-12 12-13
exact/norm bonds :
4-7 7-30 11-14 15-16 15-17 15-18 17-21 18-19 25-26
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6 8-9 8-13 9-10 10-11 11-12 12-13
isolated ring systems :
containing 1 : 8 :

G1:[*1],[*2]

Connectivity :
23:1 E exact RC ring/chain 25:2 E exact RC ring/chain
Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:Atom 9:Atom 10:Atom 11:Atom
12:Atom 13:Atom 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS 19:CLASS 21:CLASS
23:CLASS 25:CLASS 26:Atom 30:CLASS
fragments assigned product role:
containing 1
fragments assigned reactant/reagent role:

containing 8
containing 15

Search history


```
=> d his full

     (FILE 'HOME' ENTERED AT 15:22:57 ON 03 MAY 2006)

     FILE 'STNGUIDE' ENTERED AT 15:23:28 ON 03 MAY 2006

     FILE 'CASREACT' ENTERED AT 15:28:35 ON 03 MAY 2006
L1                STRUCTURE UPLOADED
L2             12 SEA SSS SAM L1 (     65 REACTIONS)

     FILE 'CAPLUS' ENTERED AT 15:29:02 ON 03 MAY 2006
L3              1 SEA ABB=ON  PLU=ON  US2003-734208/AP
                SEL RN

     FILE 'REGISTRY' ENTERED AT 15:29:24 ON 03 MAY 2006
L4             32 SEA ABB=ON  PLU=ON  (105-58-8/BI OR 10541-83-0/BI OR 110-71-4/B
                I OR 112-49-2/BI OR 118-92-3/BI OR 119-68-6/BI OR 123-30-8/BI
                OR 141814-27-9/BI OR 14703-69-6/BI OR 150-13-0/BI OR 150-75-4/B
                I OR 15022-08-9/BI OR 17318-49-9/BI OR 181819-75-0/BI OR
                214470-03-8/BI OR 2835-68-9/BI OR 29055-08-1/BI OR 3459-92-5/BI
                 OR 35472-56-1/BI OR 38359-26-1/BI OR 5344-90-1/BI OR 591-27-5/
                BI OR 611-24-5/BI OR 616-38-6/BI OR 623-04-1/BI OR 7440-23-5/BI
                 OR 7440-37-1/BI OR 7505-81-9/BI OR 7727-37-9/BI OR 88-68-6/BI
                OR 89-50-9/BI OR 95-55-6/BI)

     FILE 'CASREACT' ENTERED AT 15:29:38 ON 03 MAY 2006
L5                STRUCTURE UPLOADED
L6              6 SEA SSS SAM L5 (      6 REACTIONS)

     FILE 'REGISTRY' ENTERED AT 15:30:18 ON 03 MAY 2006
L7              6 SEA ABB=ON  PLU=ON  FAUJASIT?

     FILE 'CAPLUS' ENTERED AT 15:30:29 ON 03 MAY 2006
L8           3769 SEA ABB=ON  PLU=ON  FAUJASIT?/OBI

     FILE 'REGISTRY' ENTERED AT 15:30:44 ON 03 MAY 2006
L9              1 SEA ABB=ON  PLU=ON  ANILINE/CN

     FILE 'CAPLUS' ENTERED AT 15:30:56 ON 03 MAY 2006
L10         49252 SEA ABB=ON  PLU=ON  L9
L11            23 SEA ABB=ON  PLU=ON  L8 AND L10
L12         25354 SEA ABB=ON  PLU=ON  L9 (L) (RCT OR RGT OR RACT)/RL
L13            16 SEA ABB=ON  PLU=ON  L8 AND L12
                SEL RN L11

     FILE 'REGISTRY' ENTERED AT 15:36:53 ON 03 MAY 2006
L14           297 SEA ABB=ON  PLU=ON  (62-53-3/BI OR 1335-30-4/BI OR 100-61-8/BI
                OR 106-49-0/BI OR 121-69-7/BI OR 104-94-9/BI OR 95-53-4/BI OR
                100-01-6/BI OR 106-47-8/BI OR 108-95-2/BI OR 616-38-6/BI OR
                7631-86-9/BI OR 100-15-2/BI OR 108-44-1/BI OR 108-88-3/BI OR
                1314-23-4/BI OR 1330-20-7/BI OR 1344-28-1/BI OR 67-56-1/BI OR
                71-43-2/BI OR 99-97-8/BI OR 100-23-2/BI OR 100-41-4/BI OR
                100-46-9/BI OR 101-77-9/BI OR 101-81-5/BI OR 103-32-2/BI OR
                103-69-5/BI OR 108-67-8/BI OR 110-86-1/BI OR 12173-28-3/BI OR
                12173-98-7/BI OR 1309-48-4/BI OR 1314-35-8/BI OR 13463-67-7/BI
                OR 141814-27-9/BI OR 150-13-0/BI OR 23713-49-7/BI OR 3459-92-5/
                BI OR 50-00-0/BI OR 588-59-0/BI OR 589-16-2/BI OR 5961-59-1/BI
                OR 623-08-5/BI OR 643-28-7/BI OR 67-63-0/BI OR 7440-05-3/BI OR
                7440-06-4/BI OR 7440-50-8/BI OR 90-04-0/BI OR 932-96-7/BI OR
                98-82-8/BI OR 98-95-3/BI OR 100-00-5/BI OR 100-17-4/BI OR
```

```
                 100-42-5/BI OR 100-44-7/BI OR 100-47-0/BI OR 100-66-3/BI OR
                 10072-05-6/BI OR 102-07-8/BI OR 103-29-7/BI OR 103-65-1/BI OR
                 103-67-3/BI OR 103-73-1/BI OR 103-83-3/BI OR 104-13-2/BI OR
                 104-76-7/BI OR 10402-24-1/BI OR 105-05-5/BI OR 105-58-8/BI OR
                 10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR 106-98-9/BI OR
                 108-38-3/BI OR 108-39-4/BI OR 108-45-2/BI OR 108-47-4/BI OR
                 108-48-5/BI OR 108-89-4/BI OR 108-99-6/BI OR 109-06-8/BI OR
                 109-75-1/BI OR 110-54-3/BI OR 110-82-7/BI OR 11098-99-0/BI OR
                 11099-11-9/BI OR 111-77-3/BI OR 11116-47-5/BI OR 11118-57-3/BI
                 OR 112-49-2/BI OR 112-62-9/BI OR 115-07-1/BI OR 115-11-7/BI OR
                 119-61-9/BI OR 119-65-3/BI OR 1197-19-9/BI OR 120-12-7/BI OR
                 121-44-8/BI OR 1219-99-4/BI OR 122-39-4/BI OR 1227-44-7/BI OR
                 12417-81-1/BI OR 12510-42-8/BI OR 12627-00-8/BI OR 12737-86-9/B
                 I OR 129-00-0/BI OR 1300-71-6/BI OR 1300-73-8/BI OR 1302-42-7/B
                 I OR 1303-86-2/BI OR 1305-78-8/BI OR 1308-38-9/BI OR 1310-73-2/
                 BI OR 1311-10-0/BI OR

     FILE 'CAPLUS' ENTERED AT 15:37:35 ON 03 MAY 2006
L15         310569 SEA ABB=ON  PLU=ON  L14/P
L16         310605 SEA ABB=ON  PLU=ON  L14/PREP
L17             20 SEA ABB=ON  PLU=ON  L16 AND L11
L18         447798 SEA ABB=ON  PLU=ON  ?CARBONAT?/BI
L19              6 SEA ABB=ON  PLU=ON  L11 AND L18
                   SEL HIT RN
                   SEL HIT RN L17

     FILE 'REGISTRY' ENTERED AT 15:39:07 ON 03 MAY 2006
L20             80 SEA ABB=ON  PLU=ON  (62-53-3/BI OR 100-61-8/BI OR 121-69-7/BI
                 OR 100-15-2/BI OR 106-49-0/BI OR 95-53-4/BI OR 100-23-2/BI OR
                 101-77-9/BI OR 103-32-2/BI OR 103-69-5/BI OR 643-28-7/BI OR
                 932-96-7/BI OR 99-97-8/BI OR 100-41-4/BI OR 10072-05-6/BI OR
                 101-81-5/BI OR 102-07-8/BI OR 103-29-7/BI OR 104-94-9/BI OR
                 10541-83-0/BI OR 106-47-8/BI OR 108-44-1/BI OR 108-45-2/BI OR
                 108-47-4/BI OR 108-48-5/BI OR 108-99-6/BI OR 109-06-8/BI OR
                 1197-19-9/BI OR 1219-99-4/BI OR 122-39-4/BI OR 1227-44-7/BI OR
                 1300-73-8/BI OR 13519-75-0/BI OR 13519-80-7/BI OR 14309-92-3/BI
                  OR 150-13-0/BI OR 1943-87-9/BI OR 20642-93-7/BI OR 214470-03-8
                 /BI OR 21911-84-2/BI OR 23574-01-8/BI OR 24007-66-7/BI OR
                 24544-04-5/BI OR 2603-10-3/BI OR 28685-60-1/BI OR 2948-37-0/BI
                 OR 30438-94-9/BI OR 30448-32-9/BI OR 329767-78-4/BI OR
                 3665-80-3/BI OR 40871-06-5/BI OR 4138-15-2/BI OR 4138-40-3/BI
                 OR 42313-52-0/BI OR 4714-62-9/BI OR 53733-94-1/BI OR 56525-67-8
                 /BI OR 589-09-3/BI OR 5961-59-1/BI OR 611-21-2/BI OR 620-50-8/B
                 I OR 621-00-1/BI OR 622-14-0/BI OR 623-08-5/BI OR 698-69-1/BI
                 OR 7143-42-2/BI OR 74-90-8/BI OR 767-71-5/BI OR 768-52-5/BI OR
                 769-06-2/BI OR 793-19-1/BI OR 85-91-6/BI OR 864377-09-3/BI OR
                 90-04-0/BI OR 91-66-7/BI OR 91-73-6/BI OR 94563-11-8/BI OR
                 95-51-2/BI OR 95-55-6/BI OR 99-88-7/BI)

     FILE 'CAPLUS' ENTERED AT 15:39:42 ON 03 MAY 2006
                   SEL RN L13

     FILE 'REGISTRY' ENTERED AT 15:39:51 ON 03 MAY 2006
L21            218 SEA ABB=ON  PLU=ON  (1335-30-4/BI OR 62-53-3/BI OR 100-61-8/BI
                 OR 106-49-0/BI OR 121-69-7/BI OR 100-01-6/BI OR 104-94-9/BI OR
                 106-47-8/BI OR 616-38-6/BI OR 95-53-4/BI OR 100-15-2/BI OR
                 7631-86-9/BI OR 99-97-8/BI OR 100-23-2/BI OR 100-46-9/BI OR
                 101-77-9/BI OR 103-32-2/BI OR 108-44-1/BI OR 108-88-3/BI OR
                 110-86-1/BI OR 1309-48-4/BI OR 1314-23-4/BI OR 1314-35-8/BI OR
                 1330-20-7/BI OR 1344-28-1/BI OR 13463-67-7/BI OR 141814-27-9/BI
```

```
                    OR 3459-92-5/BI OR 50-00-0/BI OR 589-16-2/BI OR 5961-59-1/BI
                    OR 623-08-5/BI OR 643-28-7/BI OR 67-56-1/BI OR 71-43-2/BI OR
                    7440-05-3/BI OR 7440-06-4/BI OR 7440-50-8/BI OR 932-96-7/BI OR
                    100-41-4/BI OR 100-44-7/BI OR 100-47-0/BI OR 100-66-3/BI OR
                    10072-05-6/BI OR 101-81-5/BI OR 102-07-8/BI OR 103-65-1/BI OR
                    103-67-3/BI OR 103-69-5/BI OR 103-73-1/BI OR 103-83-3/BI OR
                    104-13-2/BI OR 10402-24-1/BI OR 105-05-5/BI OR 105-58-8/BI OR
                    10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR 106-98-9/BI OR
                    108-38-3/BI OR 108-39-4/BI OR 108-67-8/BI OR 108-89-4/BI OR
                    108-95-2/BI OR 109-75-1/BI OR 110-54-3/BI OR 110-82-7/BI OR
                    11098-99-0/BI OR 11099-11-9/BI OR 111-77-3/BI OR 11118-57-3/BI
                    OR 112-49-2/BI OR 115-07-1/BI OR 115-11-7/BI OR 119-65-3/BI OR
                    1197-19-9/BI OR 120-12-7/BI OR 121-44-8/BI OR 12173-28-3/BI OR
                    12173-98-7/BI OR 1219-99-4/BI OR 1227-44-7/BI OR 12627-00-8/BI
                    OR 129-00-0/BI OR 1300-71-6/BI OR 1300-73-8/BI OR 1302-42-7/BI
                    OR 1303-86-2/BI OR 1305-78-8/BI OR 1308-38-9/BI OR 1310-73-2/BI
                    OR 1311-10-0/BI OR 1314-13-2/BI OR 1314-56-3/BI OR 1314-62-1/B
                    I OR 13308-51-5/BI OR 1333-41-1/BI OR 13330-29-5/BI OR
                    1345-13-7/BI OR 135-01-3/BI OR 13519-75-0/BI OR 13519-80-7/BI
                    OR 137273-36-0/BI OR 13765-95-2/BI OR 13765-96-3/BI OR
                    141-93-5/BI OR 141-97-9/BI OR 14309-92-3/BI OR 14414-90-5/BI
                    OR 150-13-0/BI OR 15022-08-9/BI OR 156-43-4/BI OR 1864-93-3/BI
                    OR 18707-60-3/BI OR 1943-8

     FILE 'CAPLUS' ENTERED AT 15:40:21 ON 03 MAY 2006
L22        227422 SEA ABB=ON  PLU=ON  L21/PREP
L23            14 SEA ABB=ON  PLU=ON  L22 AND L13
                  SEL HIT RN

     FILE 'REGISTRY' ENTERED AT 15:40:55 ON 03 MAY 2006
L24            63 SEA ABB=ON  PLU=ON  (62-53-3/BI OR 100-61-8/BI OR 121-69-7/BI
                    OR 100-15-2/BI OR 100-23-2/BI OR 101-77-9/BI OR 103-32-2/BI OR
                    106-49-0/BI OR 643-28-7/BI OR 932-96-7/BI OR 95-53-4/BI OR
                    99-97-8/BI OR 10072-05-6/BI OR 102-07-8/BI OR 103-69-5/BI OR
                    10541-83-0/BI OR 1197-19-9/BI OR 1219-99-4/BI OR 1227-44-7/BI
                    OR 1300-73-8/BI OR 13519-75-0/BI OR 13519-80-7/BI OR 14309-92-3
                    /BI OR 1943-87-9/BI OR 20642-93-7/BI OR 214470-03-8/BI OR
                    21911-84-2/BI OR 23574-01-8/BI OR 24007-66-7/BI OR 24544-04-5/B
                    I OR 2603-10-3/BI OR 28685-60-1/BI OR 2948-37-0/BI OR 30438-94-
                    9/BI OR 30448-32-9/BI OR 329767-78-4/BI OR 3665-80-3/BI OR
                    40871-06-5/BI OR 4138-15-2/BI OR 4138-40-3/BI OR 42313-52-0/BI
                    OR 4714-62-9/BI OR 53733-94-1/BI OR 56525-67-8/BI OR 589-09-3/B
                    I OR 5961-59-1/BI OR 611-21-2/BI OR 620-50-8/BI OR 621-00-1/BI
                    OR 622-14-0/BI OR 623-08-5/BI OR 698-69-1/BI OR 7143-42-2/BI
                    OR 74-90-8/BI OR 767-71-5/BI OR 768-52-5/BI OR 769-06-2/BI OR
                    793-19-1/BI OR 85-91-6/BI OR 864377-09-3/BI OR 91-73-6/BI OR
                    94563-11-8/BI OR 99-88-7/BI)

     FILE 'CAPLUS' ENTERED AT 15:41:16 ON 03 MAY 2006
L25             4 SEA ABB=ON  PLU=ON  L23 AND L19
L26             5 SEA ABB=ON  PLU=ON  L17 AND L19
L27             5 SEA ABB=ON  PLU=ON  (L25 OR L26)

     FILE 'CASREACT' ENTERED AT 15:41:45 ON 03 MAY 2006
L28             6 SEA SSS SAM L5 (      6 REACTIONS)
L29            91 SEA SSS FUL L5 (    702 REACTIONS)
L30           179 SEA ABB=ON  PLU=ON  FAUJASIT?
L31             5 SEA ABB=ON  PLU=ON  L29 AND L30

     FILE 'CAPLUS' ENTERED AT 15:42:39 ON 03 MAY 2006
```

```
L32           5 SEA ABB=ON  PLU=ON  L31
L33           2 SEA ABB=ON  PLU=ON  L19 AND L32

     FILE 'CASREACT' ENTERED AT 15:42:57 ON 03 MAY 2006
L34             STRUCTURE UPLOADED
L35           4 SEA SUB=L29 SSS SAM L34 (     4 REACTIONS)
L36          48 SEA SUB=L29 SSS FUL L34 (   243 REACTIONS)
L37          32 SEA ABB=ON  PLU=ON  SELVA M?/AU
L38          57 SEA ABB=ON  PLU=ON  TUNDO P?/AU
L39           7 SEA ABB=ON  PLU=ON  L36 AND (L37 OR L38)

     FILE 'CAPLUS' ENTERED AT 15:44:21 ON 03 MAY 2006
L40          70 SEA ABB=ON  PLU=ON  SELVA M?/AU
L41         196 SEA ABB=ON  PLU=ON  TUNDO P?/AU
L42          62 SEA ABB=ON  PLU=ON  L40 AND L41
L43           3 SEA ABB=ON  PLU=ON  (L40 OR L41) AND (L19 OR L25 OR L26)
L44           3 SEA ABB=ON  PLU=ON  (L40 OR L41) AND (L13 OR L23)
L45          48 SEA ABB=ON  PLU=ON  L36
L46           5 SEA ABB=ON  PLU=ON  L8 AND L45
L47           5 SEA ABB=ON  PLU=ON  L32 AND L46
L48           5 SEA ABB=ON  PLU=ON  L30 AND L36

     FILE 'CASREACT' ENTERED AT 15:46:19 ON 03 MAY 2006
L49          48 SEA ABB=ON  PLU=ON  L31 OR L36 OR L48
L50          41 SEA ABB=ON  PLU=ON  L49 NOT L39

     FILE 'CAPLUS' ENTERED AT 15:47:01 ON 03 MAY 2006
L51          86 SEA ABB=ON  PLU=ON  L18 AND L8
L52           6 SEA ABB=ON  PLU=ON  L51 AND L10
L53        9721 SEA ABB=ON  PLU=ON  AMINES/CT (L) AROMATIC/OBI
L54           5 SEA ABB=ON  PLU=ON  L51 AND L53
L55           3 SEA ABB=ON  PLU=ON  L52 AND L54
L56           8 SEA ABB=ON  PLU=ON  L19 OR L25 OR L26 OR L54 OR L55
L57           4 SEA ABB=ON  PLU=ON  (L40 OR L41) AND L56

     FILE 'CASREACT' ENTERED AT 15:50:07 ON 03 MAY 2006
                D STAT QUE L31
                D STAT QUE L36
                D QUE NOS L48
L58          41 SEA ABB=ON  PLU=ON  (L31 OR L36 OR L48) NOT L39

     FILE 'REGISTRY' ENTERED AT 15:51:42 ON 03 MAY 2006

     FILE 'CAPLUS' ENTERED AT 15:51:46 ON 03 MAY 2006
                D STAT QUE L19
                D STAT QUE L25
                D STAT QUE L26
                D STAT QUE L54
                D STAT QUE L54
                D STAT QUE L55
L59           4 SEA ABB=ON  PLU=ON  (L19 OR L25 OR L26 OR L54 OR L55) NOT L57

     FILE 'CASREACT, CAPLUS' ENTERED AT 15:54:05 ON 03 MAY 2006
L60          44 DUP REM L58 L59 (1 DUPLICATE REMOVED)
                     ANSWERS '1-41' FROM FILE CASREACT
                     ANSWERS '42-44' FROM FILE CAPLUS
                D IBIB ABS HIT L60 1-41
                D IBIB ABS HITIND HITSTR L60 42-44

     FILE 'CASREACT' ENTERED AT 16:00:03 ON 03 MAY 2006
```

```
            D STAT QUE L39

     FILE 'CAPLUS' ENTERED AT 16:00:22 ON 03 MAY 2006
            D STAT QUE L57

     FILE 'CASREACT, CAPLUS' ENTERED AT 16:00:38 ON 03 MAY 2006
L61          9 DUP REM L39 L57 (2 DUPLICATES REMOVED)
                 ANSWERS '1-7' FROM FILE CASREACT
                 ANSWERS '8-9' FROM FILE CAPLUS
            D IBIB ABS HIT L61 1-7
            D IBIB ABS HITIND HITSTR L61 8-9
```

FILE HOME

FILE STNGUIDE
FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: May 2, 2006 (20060502/UP).

FILE CASREACT

FILE CONTENT:1840 - 30 Apr 2006 VOL 144 ISS 18

New CAS Information Use Policies, enter HELP USAGETERMS for details.

```
*******************************************************
*                                                      *
*     CASREACT now has more than 10 million reactions  *
*                                                      *
*******************************************************
```

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

FILE CAPLUS

FILE COVERS 1907 - 3 May 2006 VOL 144 ISS 19
FILE LAST UPDATED: 2 May 2006 (20060502/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

http://www.cas.org/infopolicy.html

FILE REGISTRY
Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES: 2 MAY 2006 HIGHEST RN 882569-16-6
DICTIONARY FILE UPDATES: 2 MAY 2006 HIGHEST RN 882569-16-6

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH January 6, 2006

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

```
*******************************************************************
*                                                                 *
* The CA roles and document type information have been removed from *
* the IDE default display format and the ED field has been added,   *
* effective March 20, 2005.  A new display format, IDERL, is now    *
* available and contains the CA role and document type information. *
*                                                                 *
*******************************************************************
```

Structure search iteration limits have been increased. See HELP SLIMITS
for details.

REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document. For information
on property searching in REGISTRY, refer to:

http://www.cas.org/ONLINE/UG/regprops.html

=>

=> file casreact
FILE 'CASREACT' ENTERED AT 15:50:07 ON 03 MAY 2006

CASREACT SEARCH

FILE CONTENT:1840 - 30 Apr 2006 VOL 144 ISS 18

New CAS Information Use Policies, enter HELP USAGETERMS for details.

```
**************************************************************
*                                                            *
*     CASREACT now has more than 10 million reactions        *
*                                                            *
**************************************************************
```

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d stat que L31
L5 STR

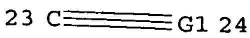
C 30 N 31

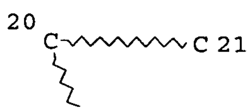
25 C M3

26 C M2

27 C M1

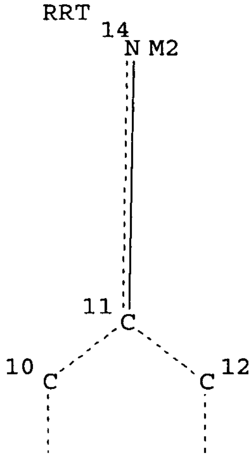
Page 1-A





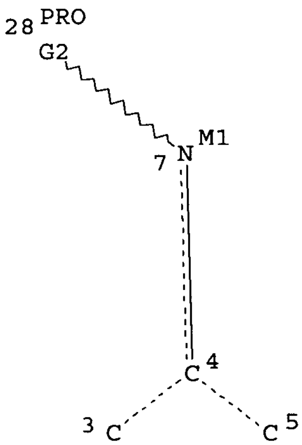
Page 1-B

RRT

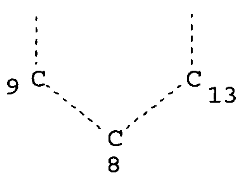


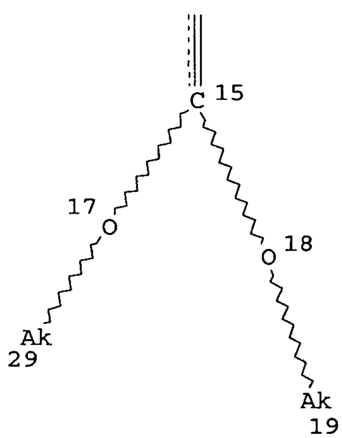
RRT

Page 2-A

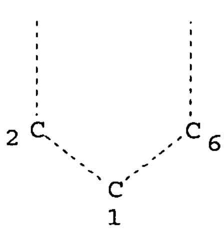


Page 2-B





Page 3-A



Page 3-B

```
VAR G1=30/31
VAR G2=20/23/25/26/27
NODE ATTRIBUTES:
HCOUNT   IS M1     AT   7
HCOUNT   IS M2     AT  14
HCOUNT   IS M3     AT  25
HCOUNT   IS M2     AT  26
HCOUNT   IS M1     AT  27
NSPEC    IS R      AT   1
NSPEC    IS R      AT   2
NSPEC    IS R      AT   3
NSPEC    IS R      AT   4
NSPEC    IS R      AT   5
NSPEC    IS R      AT   6
NSPEC    IS C      AT   7
NSPEC    IS R      AT   8
NSPEC    IS R      AT   9
NSPEC    IS R      AT  10
NSPEC    IS R      AT  11
NSPEC    IS R      AT  12
NSPEC    IS R      AT  13
NSPEC    IS C      AT  14
NSPEC    IS C      AT  15
NSPEC    IS C      AT  16
NSPEC    IS C      AT  17
NSPEC    IS C      AT  18
NSPEC    IS C      AT  19
NSPEC    IS C      AT  20
NSPEC    IS C      AT  21
NSPEC    IS C      AT  22
NSPEC    IS C      AT  23
NSPEC    IS C      AT  24
NSPEC    IS C      AT  25
NSPEC    IS C      AT  26
NSPEC    IS C      AT  27
NSPEC    IS C      AT  28
NSPEC    IS C      AT  29
DEFAULT MLEVEL IS ATOM
MLEVEL  IS CLASS  AT   7 14 15 16 17 18 19 20 21 22 23 25 26 27 29 30 31
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  31

STEREO ATTRIBUTES: NONE
L29            91 SEA FILE=CASREACT SSS FUL L5 (    702 REACTIONS)
L30           179 SEA FILE=CASREACT ABB=ON  PLU=ON  FAUJASIT?
L31             5 SEA FILE=CASREACT ABB=ON  PLU=ON  L29 AND L30


=> d stat que L36
L5              STR
```

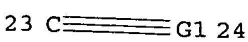
C 30 N 31

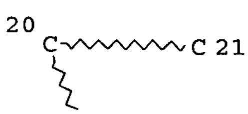
25 C M3

26 C M2

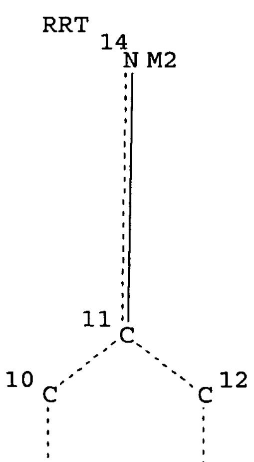
27 C M1

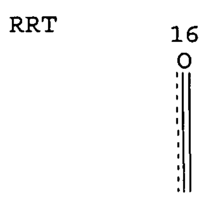
Page 1-A



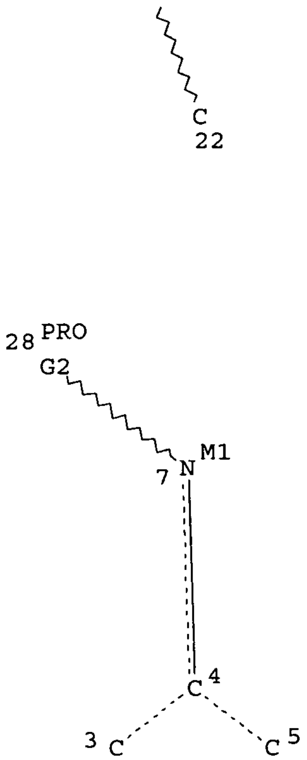


Page 1-B

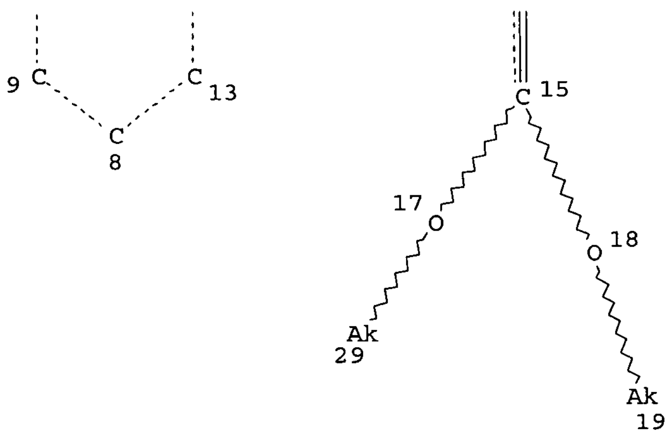




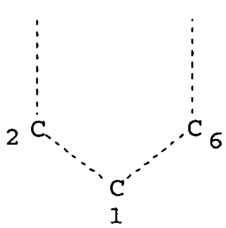
Page 2-A



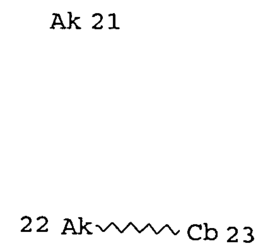
Page 2-B



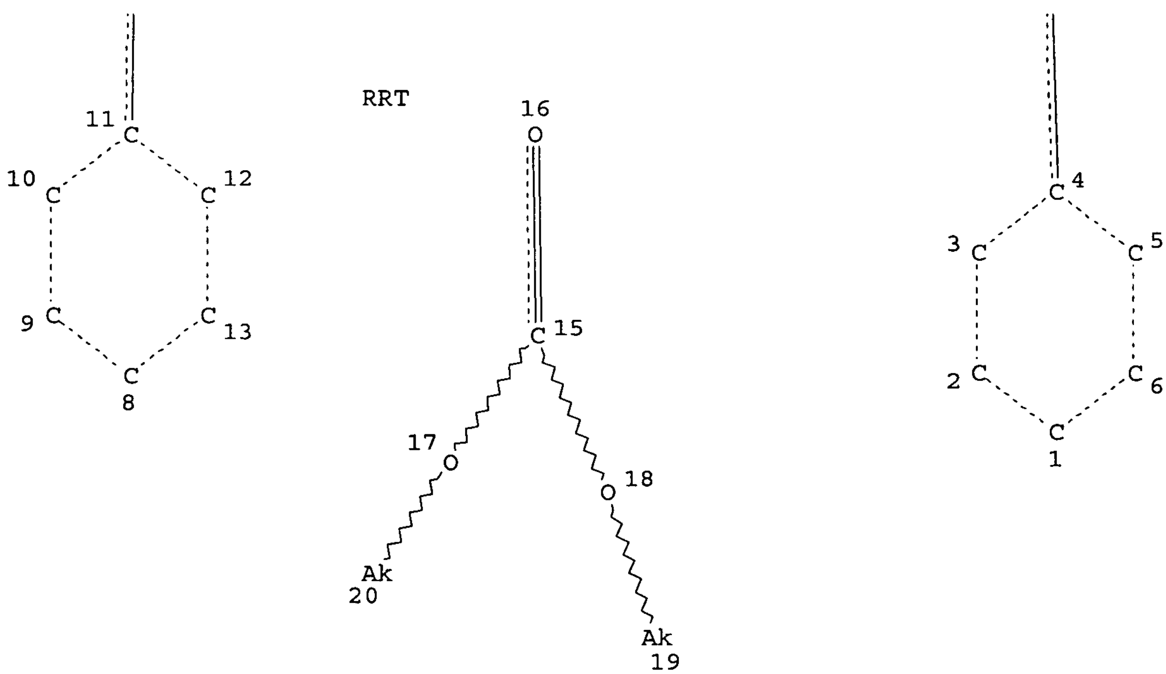
Page 3-A



```
Page 3-B
VAR G1=30/31
VAR G2=20/23/25/26/27
NODE ATTRIBUTES:
HCOUNT   IS M1      AT   7
HCOUNT   IS M2      AT  14
HCOUNT   IS M3      AT  25
HCOUNT   IS M2      AT  26
HCOUNT   IS M1      AT  27
NSPEC    IS R       AT   1
NSPEC    IS R       AT   2
NSPEC    IS R       AT   3
NSPEC    IS R       AT   4
NSPEC    IS R       AT   5
NSPEC    IS R       AT   6
NSPEC    IS C       AT   7
NSPEC    IS R       AT   8
NSPEC    IS R       AT   9
NSPEC    IS R       AT  10
NSPEC    IS R       AT  11
NSPEC    IS R       AT  12
NSPEC    IS R       AT  13
NSPEC    IS C       AT  14
NSPEC    IS C       AT  15
NSPEC    IS C       AT  16
NSPEC    IS C       AT  17
NSPEC    IS C       AT  18
NSPEC    IS C       AT  19
NSPEC    IS C       AT  20
NSPEC    IS C       AT  21
NSPEC    IS C       AT  22
NSPEC    IS C       AT  23
NSPEC    IS C       AT  24
NSPEC    IS C       AT  25
NSPEC    IS C       AT  26
NSPEC    IS C       AT  27
NSPEC    IS C       AT  28
NSPEC    IS C       AT  29
DEFAULT MLEVEL IS ATOM
MLEVEL   IS CLASS   AT   7 14 15 16 17 18 19 20 21 22 23 25 26 27 29 30 31
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  31

STEREO ATTRIBUTES: NONE
L29           91 SEA FILE=CASREACT SSS FUL L5 (    702 REACTIONS)
L34              STR
```



Page 1-A



Page 2-A
VAR G1=21/22
NODE ATTRIBUTES:

```
HCOUNT  IS M1      AT   7
HCOUNT  IS M2      AT  14
NSPEC   IS R       AT   1
NSPEC   IS R       AT   2
NSPEC   IS R       AT   3
NSPEC   IS R       AT   4
NSPEC   IS R       AT   5
NSPEC   IS R       AT   6
NSPEC   IS C       AT   7
NSPEC   IS R       AT   8
NSPEC   IS R       AT   9
NSPEC   IS R       AT  10
NSPEC   IS R       AT  11
NSPEC   IS R       AT  12
NSPEC   IS R       AT  13
NSPEC   IS C       AT  14
NSPEC   IS C       AT  15
NSPEC   IS C       AT  16
NSPEC   IS C       AT  17
NSPEC   IS C       AT  18
NSPEC   IS C       AT  19
NSPEC   IS C       AT  20
NSPEC   IS C       AT  21
NSPEC   IS C       AT  22
NSPEC   IS C       AT  23
NSPEC   IS C       AT  24
CONNECT IS E1   RC AT  21
CONNECT IS E2   RC AT  22
DEFAULT MLEVEL IS ATOM
MLEVEL  IS CLASS   AT   7 14 15 16 17 18 19 20 21 22
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  24

STEREO ATTRIBUTES: NONE
L36          48 SEA FILE=CASREACT SUB=L29 SSS FUL L34 (   243 REACTIONS)

100.0% DONE     702 VERIFIED     243 HIT RXNS                      48 DOCS
SEARCH TIME: 00.00.01



=> d que nos L48
L5                 STR
L29          91 SEA FILE=CASREACT SSS FUL L5 (   702 REACTIONS)
L30         179 SEA FILE=CASREACT ABB=ON  PLU=ON  FAUJASIT?
L34                STR
L36          48 SEA FILE=CASREACT SUB=L29 SSS FUL L34 (   243 REACTIONS)
L48           5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L30 AND L36

=> s (L31 or L36 or L48) not L39
L58         41 (L31 OR L36 OR L48) NOT L39
```

printed with author search

```
=> file registry
FILE 'REGISTRY' ENTERED AT 15:51:42 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
```

Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES: 2 MAY 2006 HIGHEST RN 882569-16-6
DICTIONARY FILE UPDATES: 2 MAY 2006 HIGHEST RN 882569-16-6

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH January 6, 2006

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

```
*******************************************************************
*                                                                 *
* The CA roles and document type information have been removed from *
* the IDE default display format and the ED field has been added,   *
* effective March 20, 2005.  A new display format, IDERL, is now    *
* available and contains the CA role and document type information. *
*                                                                 *
*******************************************************************
```

Structure search iteration limits have been increased. See HELP SLIMITS
for details.

REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document. For information
on property searching in REGISTRY, refer to:

http://www.cas.org/ONLINE/UG/regprops.html

=> file caplus
FILE 'CAPLUS' ENTERED AT 15:51:46 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

CAPLUS
TEXT SEARCH

FILE COVERS 1907 - 3 May 2006 VOL 144 ISS 19
FILE LAST UPDATED: 2 May 2006 (20060502/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply.
They are available for your review at:

http://www.cas.org/infopolicy.html
'OBI' IS DEFAULT SEARCH FIELD FOR 'CAPLUS' FILE

```
=> d stat que L19
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L9             1 SEA FILE=REGISTRY ABB=ON  PLU=ON  ANILINE/CN
L10        49252 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9
L11           23 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L10
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L19            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L11 AND L18


=> d stat que L25
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L9             1 SEA FILE=REGISTRY ABB=ON  PLU=ON  ANILINE/CN
L10        49252 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9
L11           23 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L10
L12        25354 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9 (L) (RCT OR RGT OR RACT)/RL

L13           16 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L12
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L19            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L11 AND L18
L21          218 SEA FILE=REGISTRY ABB=ON  PLU=ON  (1335-30-4/BI OR 62-53-3/BI
                 OR 100-61-8/BI OR 106-49-0/BI OR 121-69-7/BI OR 100-01-6/BI OR
                 104-94-9/BI OR 106-47-8/BI OR 616-38-6/BI OR 95-53-4/BI OR
                 100-15-2/BI OR 7631-86-9/BI OR 99-97-8/BI OR 100-23-2/BI OR
                 100-46-9/BI OR 101-77-9/BI OR 103-32-2/BI OR 108-44-1/BI OR
                 108-88-3/BI OR 110-86-1/BI OR 1309-48-4/BI OR 1314-23-4/BI OR
                 1314-35-8/BI OR 1330-20-7/BI OR 1344-28-1/BI OR 13463-67-7/BI
                 OR 141814-27-9/BI OR 3459-92-5/BI OR 50-00-0/BI OR 589-16-2/BI
                 OR 5961-59-1/BI OR 623-08-5/BI OR 643-28-7/BI OR 67-56-1/BI OR
                 71-43-2/BI OR 7440-05-3/BI OR 7440-06-4/BI OR 7440-50-8/BI OR
                 932-96-7/BI OR 100-41-4/BI OR 100-44-7/BI OR 100-47-0/BI OR
                 100-66-3/BI OR 10072-05-6/BI OR 101-81-5/BI OR 102-07-8/BI OR
                 103-65-1/BI OR 103-67-3/BI OR 103-69-5/BI OR 103-73-1/BI OR
                 103-83-3/BI OR 104-13-2/BI OR 10402-24-1/BI OR 105-05-5/BI OR
                 105-58-8/BI OR 10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR
                 106-98-9/BI OR 108-38-3/BI OR 108-39-4/BI OR 108-67-8/BI OR
                 108-89-4/BI OR 108-95-2/BI OR 109-75-1/BI OR 110-54-3/BI OR
                 110-82-7/BI OR 11098-99-0/BI OR 11099-11-9/BI OR 111-77-3/BI
                 OR 11118-57-3/BI OR 112-49-2/BI OR 115-07-1/BI OR 115-11-7/BI
                 OR 119-65-3/BI OR 1197-19-9/BI OR 120-12-7/BI OR 121-44-8/BI
                 OR 12173-28-3/BI OR 12173-98-7/BI OR 1219-99-4/BI OR 1227-44-7/
                 BI OR 12627-00-8/BI OR 129-00-0/BI OR 1300-71-6/BI OR 1300-73-8
                 /BI OR 1302-42-7/BI OR 1303-86-2/BI OR 1305-78-8/BI OR
                 1308-38-9/BI OR 1310-73-2/BI OR 1311-10-0/BI OR 1314-13-2/BI
                 OR 1314-56-3/BI OR 1314-62-1/BI OR 13308-51-5/BI OR 1333-41-1/B
                 I OR 13330-29-5/BI OR 1345-13-7/BI OR 135-01-3/BI OR 13519-75-0
                 /BI OR 13519-80-7/BI OR 137273-36-0/BI OR 13765-95-2/BI OR
                 13765-96-3/BI OR 141-93-5/BI OR 141-97-9/BI OR 14309-92-3/BI
                 OR 14414-90-5/BI OR 150-13-0/BI OR 15022-08-9/BI OR 156-43-4/BI
                  OR 1864-93-3/BI OR 18707-60-3/BI OR 1943-8
L22       227422 SEA FILE=CAPLUS ABB=ON  PLU=ON  L21/PREP
L23           14 SEA FILE=CAPLUS ABB=ON  PLU=ON  L22 AND L13
L25            4 SEA FILE=CAPLUS ABB=ON  PLU=ON  L23 AND L19


=> d stat que L26
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L9             1 SEA FILE=REGISTRY ABB=ON  PLU=ON  ANILINE/CN
L10        49252 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9
L11           23 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L10
L14          297 SEA FILE=REGISTRY ABB=ON  PLU=ON  (62-53-3/BI OR 1335-30-4/BI
```

```
                OR 100-61-8/BI OR 106-49-0/BI OR 121-69-7/BI OR 104-94-9/BI OR
                95-53-4/BI OR 100-01-6/BI OR 106-47-8/BI OR 108-95-2/BI OR
                616-38-6/BI OR 7631-86-9/BI OR 100-15-2/BI OR 108-44-1/BI OR
                108-88-3/BI OR 1314-23-4/BI OR 1330-20-7/BI OR 1344-28-1/BI OR
                67-56-1/BI OR 71-43-2/BI OR 99-97-8/BI OR 100-23-2/BI OR
                100-41-4/BI OR 100-46-9/BI OR 101-77-9/BI OR 101-81-5/BI OR
                103-32-2/BI OR 103-69-5/BI OR 108-67-8/BI OR 110-86-1/BI OR
                12173-28-3/BI OR 12173-98-7/BI OR 1309-48-4/BI OR 1314-35-8/BI
                OR 13463-67-7/BI OR 141814-27-9/BI OR 150-13-0/BI OR 23713-49-7
                /BI OR 3459-92-5/BI OR 50-00-0/BI OR 588-59-0/BI OR 589-16-2/BI
                 OR 5961-59-1/BI OR 623-08-5/BI OR 643-28-7/BI OR 67-63-0/BI
                OR 7440-05-3/BI OR 7440-06-4/BI OR 7440-50-8/BI OR 90-04-0/BI
                OR 932-96-7/BI OR 98-82-8/BI OR 98-95-3/BI OR 100-00-5/BI OR
                100-17-4/BI OR 100-42-5/BI OR 100-44-7/BI OR 100-47-0/BI OR
                100-66-3/BI OR 10072-05-6/BI OR 102-07-8/BI OR 103-29-7/BI OR
                103-65-1/BI OR 103-67-3/BI OR 103-73-1/BI OR 103-83-3/BI OR
                104-13-2/BI OR 104-76-7/BI OR 10402-24-1/BI OR 105-05-5/BI OR
                105-58-8/BI OR 10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR
                106-98-9/BI OR 108-38-3/BI OR 108-39-4/BI OR 108-45-2/BI OR
                108-47-4/BI OR 108-48-5/BI OR 108-89-4/BI OR 108-99-6/BI OR
                109-06-8/BI OR 109-75-1/BI OR 110-54-3/BI OR 110-82-7/BI OR
                11098-99-0/BI OR 11099-11-9/BI OR 111-77-3/BI OR 11116-47-5/BI
                OR 11118-57-3/BI OR 112-49-2/BI OR 112-62-9/BI OR 115-07-1/BI
                OR 115-11-7/BI OR 119-61-9/BI OR 119-65-3/BI OR 1197-19-9/BI
                OR 120-12-7/BI OR 121-44-8/BI OR 1219-99-4/BI OR 122-39-4/BI
                OR 1227-44-7/BI OR 12417-81-1/BI OR 12510-42-8/BI OR 12627-00-8
                /BI OR 12737-86-9/BI OR 129-00-0/BI OR 1300-71-6/BI OR
                1300-73-8/BI OR 1302-42-7/BI OR 1303-86-2/BI OR 1305-78-8/BI
                OR 1308-38-9/BI OR 1310-73-2/BI OR 1311-10-0/BI OR
L16       310605 SEA FILE=CAPLUS ABB=ON  PLU=ON  L14/PREP
L17           20 SEA FILE=CAPLUS ABB=ON  PLU=ON  L16 AND L11
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L19            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L11 AND L18
L26            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L17 AND L19


=> d stat que L54
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L51           86 SEA FILE=CAPLUS ABB=ON  PLU=ON  L18 AND L8
L53         9721 SEA FILE=CAPLUS ABB=ON  PLU=ON  AMINES/CT (L) AROMATIC/OBI
L54            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L53


=> d stat que L54
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L51           86 SEA FILE=CAPLUS ABB=ON  PLU=ON  L18 AND L8
L53         9721 SEA FILE=CAPLUS ABB=ON  PLU=ON  AMINES/CT (L) AROMATIC/OBI
L54            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L53


=> d stat que L55
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L9             1 SEA FILE=REGISTRY ABB=ON  PLU=ON  ANILINE/CN
L10        49252 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L51           86 SEA FILE=CAPLUS ABB=ON  PLU=ON  L18 AND L8
L52            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L10
L53         9721 SEA FILE=CAPLUS ABB=ON  PLU=ON  AMINES/CT (L) AROMATIC/OBI
```

```
L54            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L53
L55            3 SEA FILE=CAPLUS ABB=ON  PLU=ON  L52 AND L54


=> s (L19 or L25 or L26 or L54 or L55) not L57
L59            4 (L19 OR L25 OR L26 OR L54 OR L55) NOT L57
```

printed with author search

```
=> dup rem L58 L59
FILE 'CASREACT' ENTERED AT 15:54:05 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

FILE 'CAPLUS' ENTERED AT 15:54:05 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)
PROCESSING COMPLETED FOR L58
PROCESSING COMPLETED FOR L59
L60           44 DUP REM L58 L59 (1 DUPLICATE REMOVED)
                 ANSWERS '1-41' FROM FILE CASREACT
                 ANSWERS '42-44' FROM FILE CAPLUS

=> d ibib abs hit L60 1-41; d ibib abs hitind hitstr L60 42-44
```

L60 ANSWER 1 OF 44 CASREACT COPYRIGHT 2006 ACS on STN DUPLICATE 1

ACCESSION NUMBER: 141:395224 CASREACT

TITLE: Zeolite-promoted selective mono-N-methylation of aniline with dimethyl carbonate

AUTHOR(S): Esakkidurai, Thirugnanasamy; Pitchumani, Kasi

CORPORATE SOURCE: School of Chemistry, Madurai Kamaraj University, Madurai, 625021, India

SOURCE: Journal of Molecular Catalysis A: Chemical (2004), 218(2), 197-201
CODEN: JMCCF2; ISSN: 1381-1169

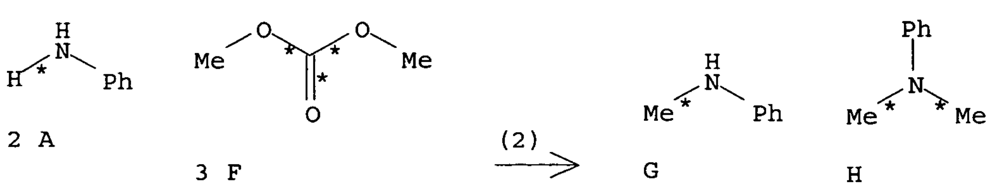
PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Selective mono-N-methylation of aniline with di-Me carbonate is carried out over **faujasite** Y zeolites. This one pot protocol is highly selective, favoring mono-N-methylation. Benzylation with dibenzyl carbonate is also found to be very selective, resulting in exclusive monobenzylation. Other advantages such as lower temperature, less rigorous reaction conditions, absence of C-alkylation products, etc., are also highlighted.

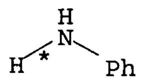
RX(2) OF 9 2 **A** + 3 **F** ===> **G** + H



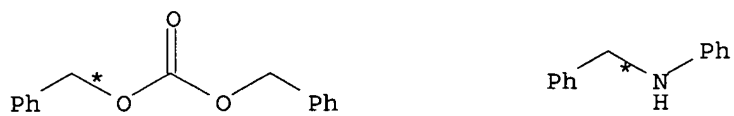
RX(2) RCT A **62-53-3**, F **616-38-6**
PRO G **100-61-8**, H 121-69-7

SOL 71-43-2 Benzene
CON 205 minutes, 100 deg C
NTE 98% conversion observed, 99:1 mono:di, Zeolite KY used as catalyst, conversion and mono-/di-selectivity depend on zeolite and rection time, green chem.-catalyst, green chem.-waste redn., no reaction observed in the absence of zeolite

RX(3) OF 9 **A + I ===> C**



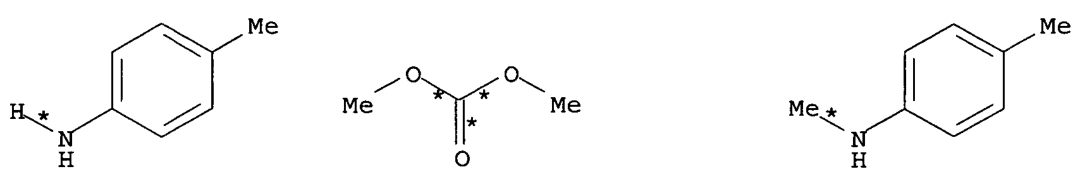
A



I (3) C

RX(3) RCT A **62-53-3**, I **3459-92-5**
PRO C **103-32-2**
SOL 71-43-2 Benzene
CON 100 deg C
NTE 100:0 mono:di, 77% conversion observed, Zeolite NaX used as catalyst, conversion depends on zeolite and solvent, green chem.-catalyst, green chem.-waste redn.

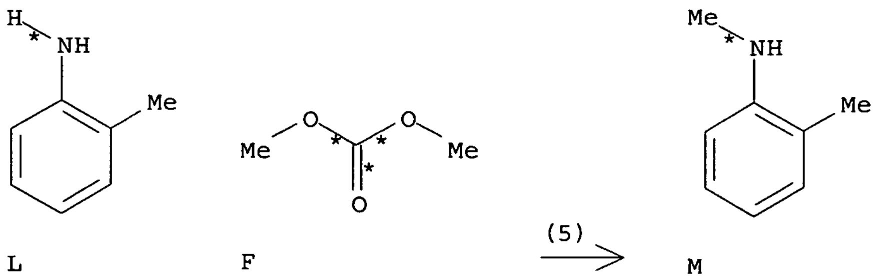
RX(4) OF 9 **J + F ===> K**



J F (4) K
YIELD 6%

RX(4) RCT J **106-49-0**, F **616-38-6**
PRO K **623-08-5**
SOL 71-43-2 Benzene
CON 205 minutes, 100 deg C
NTE Zeolite KY used as catalyst, green chem.-catalyst, green chem.-waste redn.

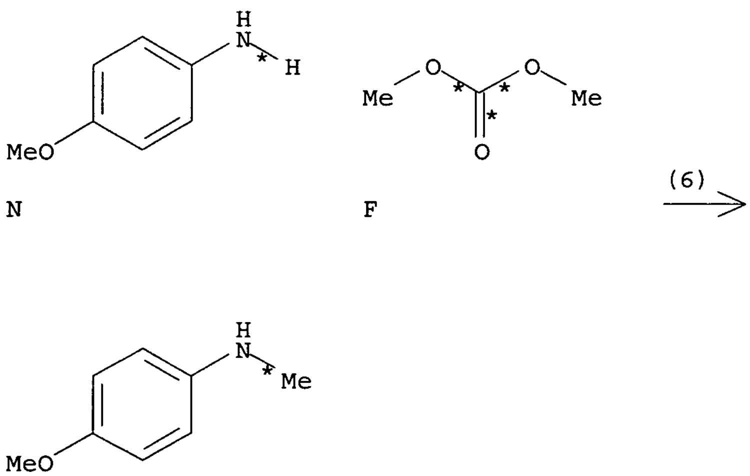
RX(5) OF 9 **L + F ===> M**



L F M
YIELD 6%

RX(5) RCT L **95-53-4**, F **616-38-6**
PRO M **611-21-2**
SOL 71-43-2 Benzene
CON 205 minutes, 100 deg C
NTE Zeolite KY used as catalyst, green chem.-catalyst, green chem.-waste redn.

RX(6) OF 9 **N + F ===> O**

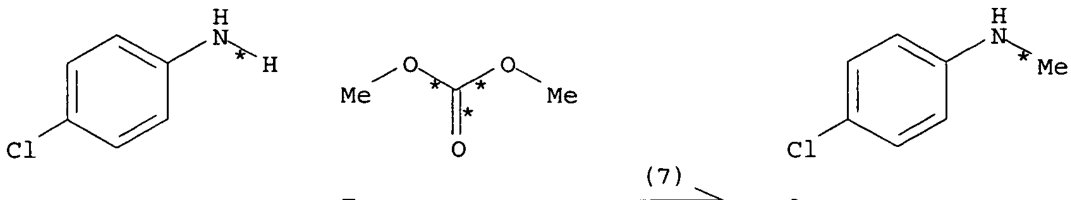


N F

O
YIELD 6%

RX(6) RCT N **104-94-9**, F **616-38-6**
PRO O **5961-59-1**
SOL 71-43-2 Benzene
CON 205 minutes, 100 deg C
NTE Zeolite KY used as catalyst, green chem.-catalyst, green chem.-waste redn.

RX(7) OF 9 **P + F ===> Q**

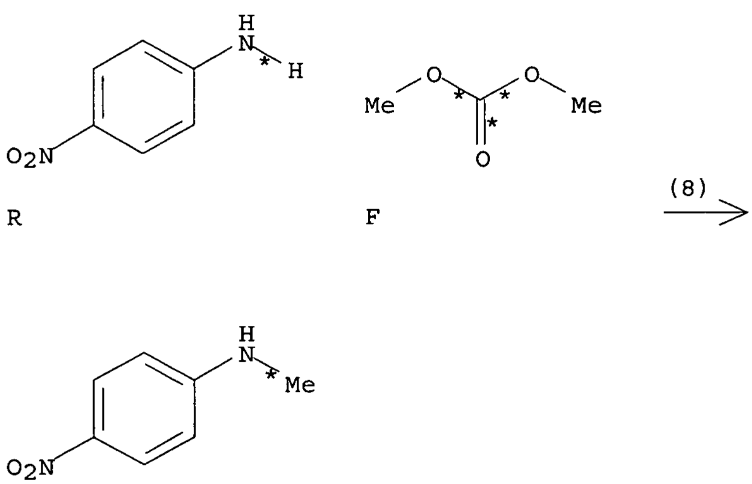


P F Q

YIELD 6%

```
RX(7)      RCT  P 106-47-8, F 616-38-6
           PRO  Q 932-96-7
           SOL  71-43-2 Benzene
           CON  205 minutes, 100 deg C
           NTE  Zeolite KY used as catalyst, green chem.-catalyst, green
                chem.-waste redn.
```

RX(8) OF 9 **R + F ===> S**



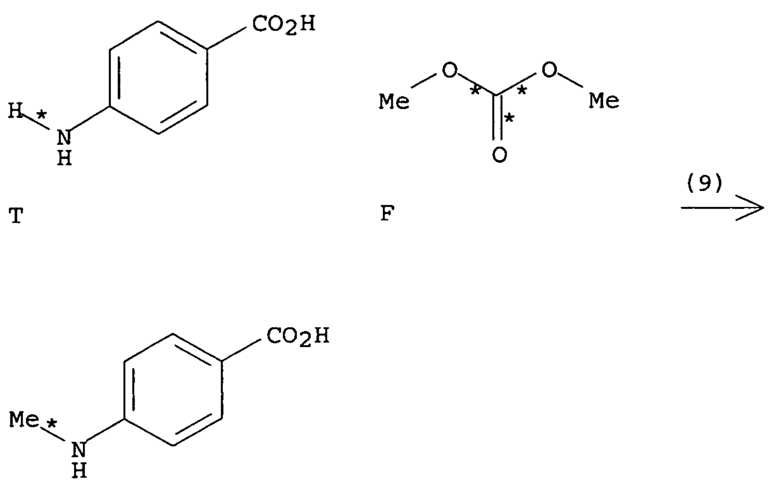
R F

S

YIELD 6%

```
RX(8)      RCT  R 100-01-6, F 616-38-6
           PRO  S 100-15-2
           SOL  71-43-2 Benzene
           CON  205 minutes, 100 deg C
           NTE  Zeolite KY used as catalyst, green chem.-catalyst, green
                chem.-waste redn.
```

RX(9) OF 9 **T + F ===> U**



T F U

YIELD 6%

RX(9) RCT T **150-13-0**, F **616-38-6**
PRO U **10541-83-0**
SOL 71-43-2 Benzene
CON 205 minutes, 100 deg C
NTE Zeolite KY used as catalyst, green chem.-catalyst, green chem.-waste redn.

AB Selective mono-N-methylation of aniline with di-Me carbonate is carried out over **faujasite** Y zeolites. This one pot protocol is highly selective, favoring mono-N-methylation. Benzylation with dibenzyl carbonate is also found to be very selective, resulting in exclusive monobenzylation. Other advantages such as lower temperature, less rigorous reaction conditions, absence of C-alkylation products, etc., are also highlighted.

ST aniline selective methylation benzylation; **faujasite** Y zeolite selective methylation benzylation catalyst; methylaniline benzylaniline prepn

L60 ANSWER 2 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 143:229795 CASREACT

TITLE: (1H-Imidazo[4,5-c]pyridin-2-yl)-1,2,5-oxadiazol-3-ylamine derivatives: A novel class of potent MSK-1-inhibitors

AUTHOR(S): Bamford, Mark J.; Alberti, Michael J.; Bailey, Nicholas; Davies, Susannah; Dean, David K.; Gaiba, Alessandra; Garland, Stephen; Harling, John D.; Jung, David K.; Panchal, Terence A.; Parr, Christopher A.; Steadman, Jon G.; Takle, Andrew K.; Townsend, James T.; Wilson, David M.; Witherington, Jason

CORPORATE SOURCE: Neurology and GI Centre of Excellence for Drug Discovery, New Frontiers Science Park, GlaxoSmithKline Pharmaceuticals, Essex, CM19 5AW, UK

SOURCE: Bioorganic & Medicinal Chemistry Letters (2005), 15(14), 3402-3406
CODEN: BMCLE8; ISSN: 0960-894X

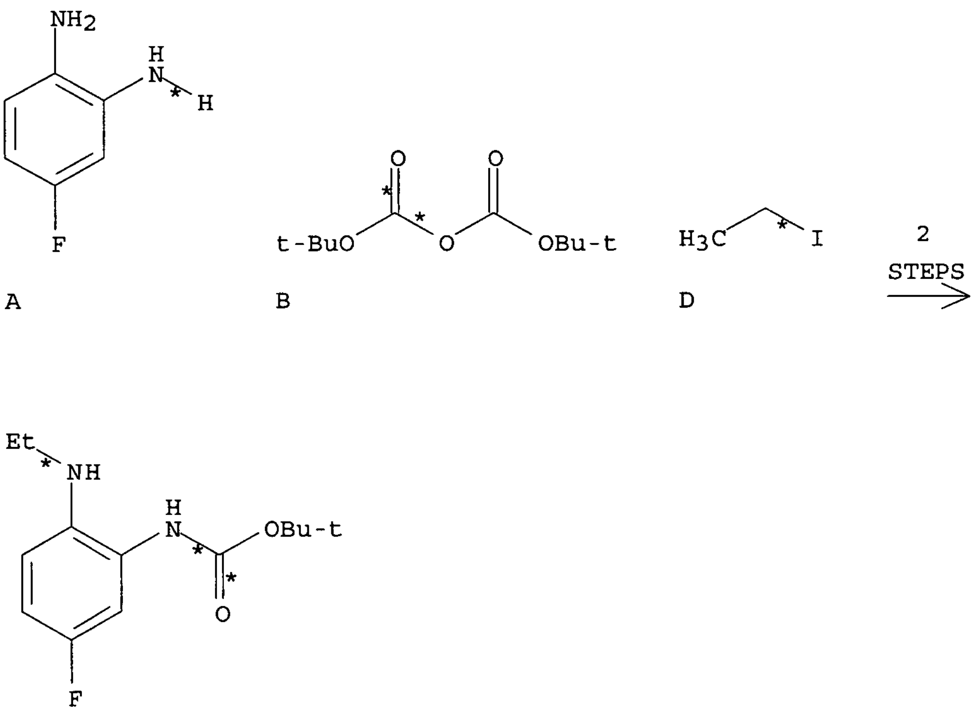
PUBLISHER: Elsevier B.V.

DOCUMENT TYPE: Journal
LANGUAGE: English

AB A novel series of imidazo[4,5-c]pyridines bearing a 1,2,5-oxadiazol-3-ylamine functionality has been developed. These are potent inhibitors of mitogen and stress-activated protein kinase-1.

REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(28) OF 60 COMPOSED OF RX(1), RX(2)
RX(28) **A** + **B** + D ===> **E**



E
YIELD 38%

RX(1) RCT A **367-31-7**, B **24424-99-5**
PRO C 362670-07-3
CON 50 deg C
NTE regioselective

RX(2) RCT C 362670-07-3

STAGE(1)
RGT F 865-47-4 t-BuOK
SOL 109-99-9 THF
CON 21 deg C

STAGE(2)
RCT D 75-03-6
CON 21 deg C

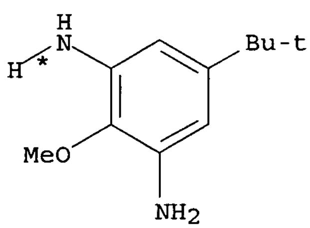
PRO E **862815-01-8**

L60 ANSWER 3 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 143:59953 CASREACT
TITLE: Exhaustively Methylated Azacalix[4]arene: Preparation, Conformation, and Crystal Structure with Exclusively CH/π-Controlled Crystal Architecture
AUTHOR(S): Tsue, Hirohito; Ishibashi, Koichi; Takahashi, Hiroki; Tamura, Rui
CORPORATE SOURCE: Graduate School of Global Environmental Studies and Graduate School of Human and Environmental Studies, Kyoto University, Kyoto, 606-8501, Japan
SOURCE: Organic Letters (2005), 7(11), 2165-2168
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

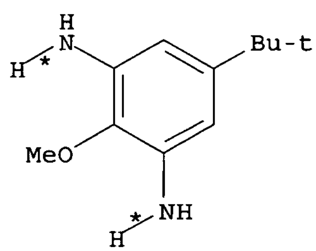
AB The preparation, conformation, and crystal structure of exhaustively methylated azacalix[4]arene involving nitrogen atoms as bridging units are described. NMR and X-ray crystallog. anal. have demonstrated that this novel azacalix[4]arene adopts a 1,3-alternate conformation both in solution and in the solid state. The crystal structure has been characterized solely by intermol. CH/π interactions, by which the azacalix[4]arenes mutually interact with each other outside the cavity to furnish a two-dimensional network structure.

REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

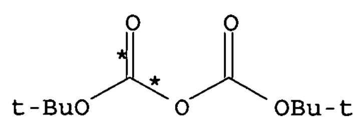
RX(9) OF 19 COMPOSED OF RX(2), RX(3)
RX(9) 2 **B** + 2 **H** + 2 L ===> **M**



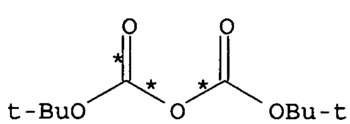
B



B



H

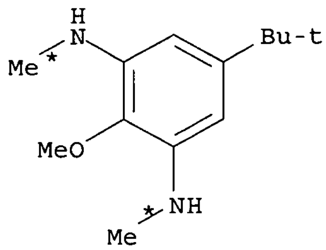


H

H_3C-*-I

2 L

2 STEPS →



M
YIELD 72%

RX(2)   RCT B **473269-70-4**, H **24424-99-5**
        PRO I 854669-26-4, J 854669-30-0
        SOL 75-65-0 t-BuOH
        CON 3 hours, room temperature

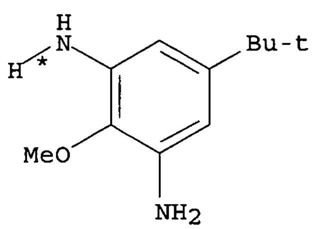
RX(3)   RCT J 854669-30-0

  STAGE(1)
    RGT N 7646-69-7 NaH
    SOL 68-12-2 DMF
    CON 10 minutes, 0 deg C

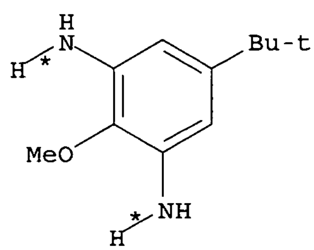
  STAGE(2)
    RCT L 74-88-4
    CON SUBSTAGE(1) 0 deg C
         SUBSTAGE(2) overnight, room temperature

        PRO M **854669-37-7**

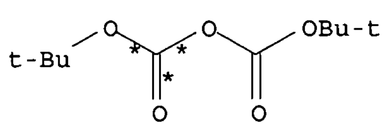
RX(14) OF 19 COMPOSED OF RX(2), RX(4), RX(5)
RX(14)   2 **B** + 2 **H** + C + 3 L ===> **Q**



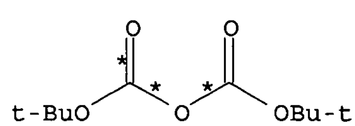
B



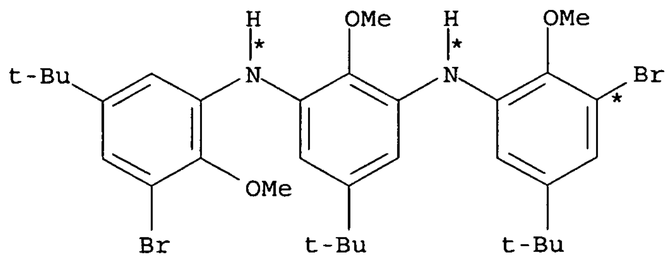
B



H



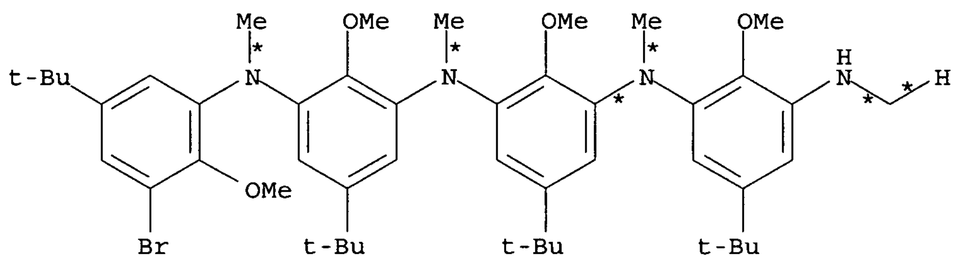
H



C

H_3C-I 3 L

3 STEPS →



Q
YIELD 81%

RX(2)	RCT	B **473269-70-4**, H **24424-99-5**
	PRO	I 854669-26-4, J 854669-30-0
	SOL	75-65-0 t-BuOH
	CON	3 hours, room temperature

RX(4)	RCT	C 854669-22-0, I 854669-26-4
	RGT	D 865-48-5 NaOBu-t
	PRO	P 854669-43-5
	CAT	166330-10-5 Phosphine, (oxydi-2,1-phenylene)bis[diphenyl-,
		3375-31-3 Pd(OAc)2
	SOL	108-88-3 PhMe
	CON	22 hours, 80 deg C

RX(5)	RCT	P 854669-43-5

STAGE(1)
	RGT	N 7646-69-7 NaH
	SOL	68-12-2 DMF
	CON	10 minutes, 0 deg C

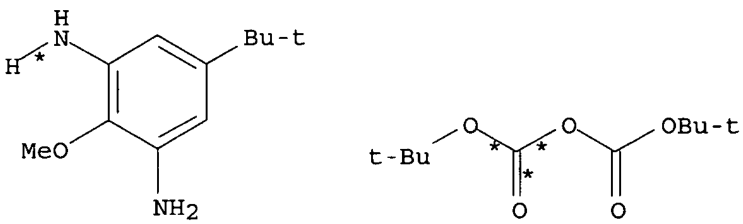
STAGE(2)
	RCT	L 74-88-4
	CON	SUBSTAGE(1) 0 deg C
		SUBSTAGE(2) overnight, room temperature

	PRO	Q **854669-49-1**

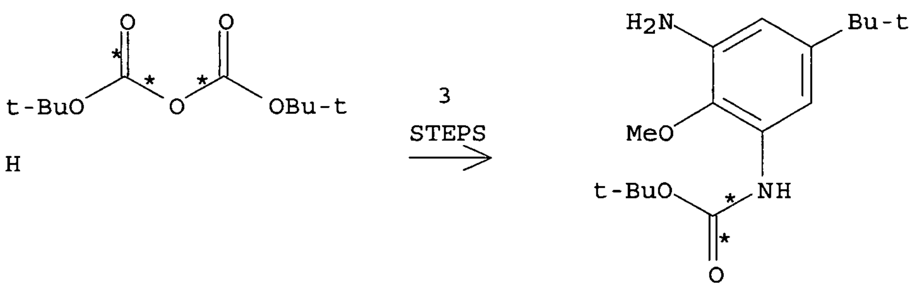
RX(18) OF 19 COMPOSED OF REACTION SEQUENCE RX(2), RX(4), RX(5)
AND REACTION SEQUENCE RX(1), RX(4), RX(5)

...2 **B** + 2 **H** ===> I...

...2 A + **B** + I + 3 L ===> **Q**



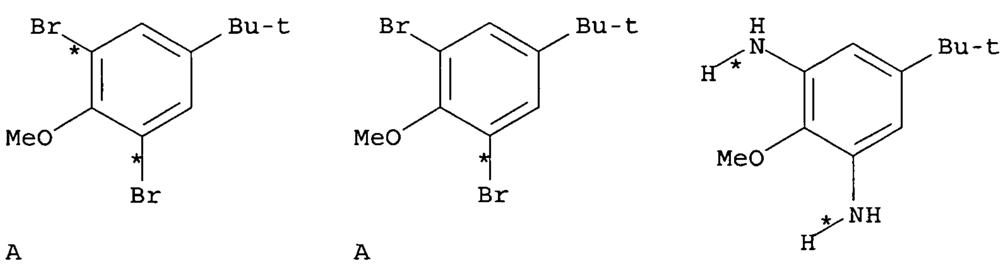
B H



H

I

START NEXT REACTION SEQUENCE



A A 2 B

```
STAGE(2)
   RCT  L 74-88-4
   CON  SUBSTAGE(1) 0 deg C
        SUBSTAGE(2) overnight, room temperature

PRO  Q 854669-49-1
```

L60 ANSWER 4 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 142:411301 CASREACT

TITLE: Inhibition of secretory phospholipase A2. 2-Synthesis and structure-activity relationship studies of 4,5-dihydro-3-(4-tetradecyloxybenzyl)-1,2,4-4H-oxadiazol-5-one (PMS1062) derivatives specific for group II enzyme

AUTHOR(S): Dong, Chang-Zhi; Ahamada-Himidi, Azali; Plocki, Stephanie; Aoun, Darina; Touaibia, Mohamed; Meddad-Bel Habich, Nadia; Huet, Jack; Redeuilh, Catherine; Ombetta, Jean-Edouard; Godfroid, Jean-Jacques; Massicot, France; Heymans, Francoise

CORPORATE SOURCE: Unite de Pharmacochimie Moleculaire et Systemes Membranaires (EA2381), Laboratoire de Pharmacochimie Moleculaire, Universite Paris 7-Denis Diderot, Paris, 75251, Fr.

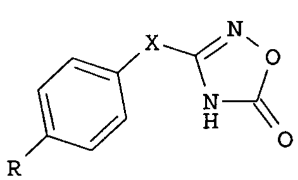
SOURCE: Bioorganic & Medicinal Chemistry (2005), 13(6), 1989-2007
CODEN: BMECEP; ISSN: 0968-0896

PUBLISHER: Elsevier Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

GI



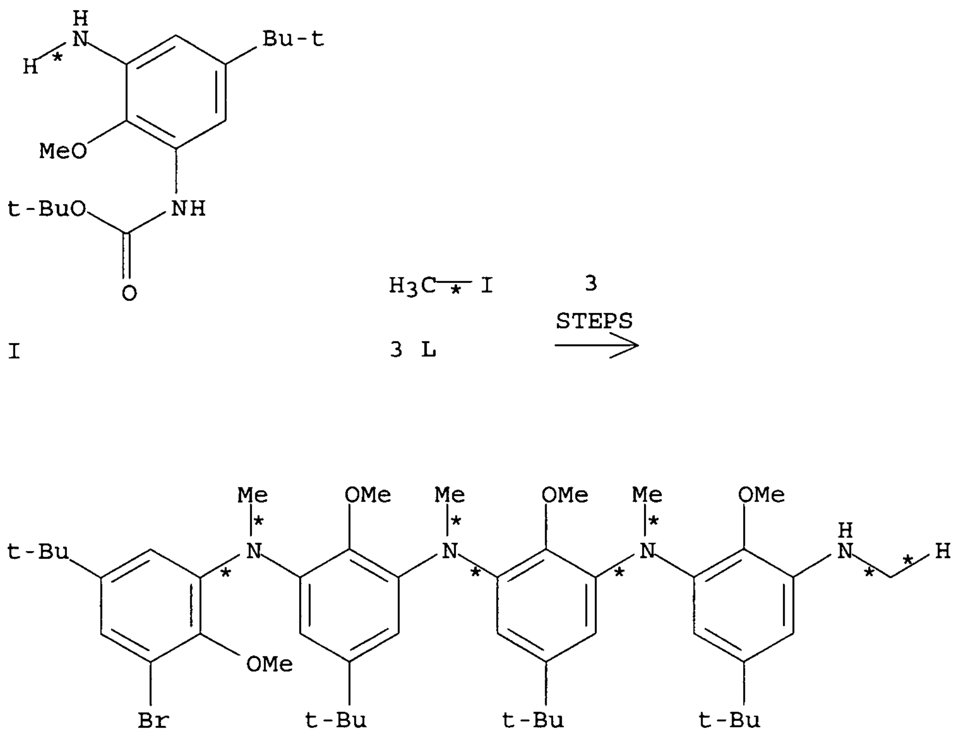
I

AB The discovery of a series of specific inhibitors of human group IIA phospholipase A2 (hGIIA PLA2) displaying promising in vitro and in vivo properties has been recently reported. Here the influence of different structural modifications on the specificity and potency of oxadiazolones, e.g. I [X = CH2, CH2CH2, CHMe, CMe2; R = MeO, n-octyloxy, n-tetradecylthio, N,N-di(heptyl)amino, etc.], against hGIIA PLA2 vs. porcine group IB PLA2 is described. The SAR results, as well as the log P and pKa values of the oxadiazolones studied provide important information towards the comprehension of the mode of action of this kind of compds.

REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(226) OF 226 COMPOSED OF RX(29), RX(30), RX(50), RX(74), RX(75)

RX(226) 3 W + 2 **BW** + **CB** + DD ===> **ED**



Q
YIELD 81%

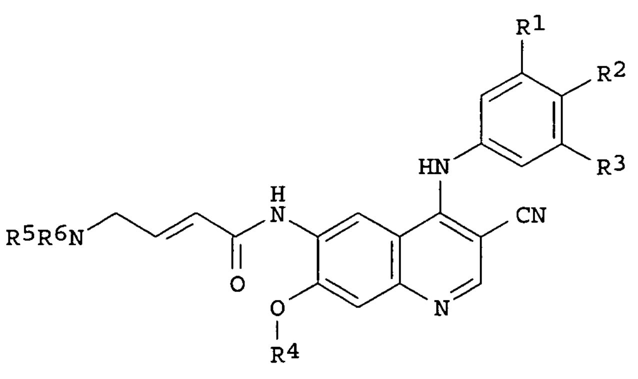
```
RX(2)      RCT  B 473269-70-4, H 24424-99-5
           PRO  I 854669-26-4, J 854669-30-0
           SOL  75-65-0 t-BuOH
           CON  3 hours, room temperature

RX(1)      RCT  A 132268-08-7, B 473269-70-4
           RGT  D 865-48-5 NaOBu-t
           PRO  C 854669-22-0
           CAT  166330-10-5 Phosphine, (oxydi-2,1-phenylene)bis[diphenyl-,
                3375-31-3 Pd(OAc)2
           SOL  108-88-3 PhMe
           CON  24 hours, 80 deg C

RX(4)      RCT  C 854669-22-0, I 854669-26-4
           RGT  D 865-48-5 NaOBu-t
           PRO  P 854669-43-5
           CAT  166330-10-5 Phosphine, (oxydi-2,1-phenylene)bis[diphenyl-,
                3375-31-3 Pd(OAc)2
           SOL  108-88-3 PhMe
           CON  22 hours, 80 deg C

RX(5)      RCT  P 854669-43-5

             STAGE(1)
                RGT  N 7646-69-7 NaH
                SOL  68-12-2 DMF
                CON  10 minutes, 0 deg C
```

STAGE(2)
SOL 108-88-3 PhMe
CON 5 hours, reflux

PRO EC 850143-73-6

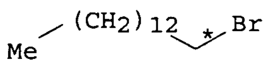
RX(75) RCT EC 850143-73-6
RGT U 7647-01-0 HCl
PRO ED **850143-74-7**
SOL 60-29-7 Et2O, 109-99-9 THF
CON SUBSTAGE(1) 2 hours, 0 deg C
SUBSTAGE(2) 1 hour, room temperature

L60 ANSWER 5 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 142:316675 CASREACT
TITLE: Optimization of 6,7-Disubstituted-4-(arylamino)quinoline-3-carbonitriles as Orally Active, Irreversible Inhibitors of Human Epidermal Growth Factor Receptor-2 Kinase Activity
AUTHOR(S): Tsou, Hwei-Ru; Overbeek-Klumpers, Elsebe G.; Hallett, William A.; Reich, Marvin F.; Floyd, M. Brawner; Johnson, Bernard D.; Michalak, Ronald S.; Nilakantan, Ramaswamy; Discafani, Carolyn; Golas, Jonathan; Rabindran, Sridhar K.; Shen, Ru; Shi, Xiaoqing; Wang, Yu-Fen; Upeslacis, Janis; Wissner, Allan
CORPORATE SOURCE: Chemical and Screening Sciences, Chemical Development, and Oncology, Wyeth Research, Pearl River, NY, 10965, USA
SOURCE: Journal of Medicinal Chemistry (2005), 48(4), 1107-1131
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
GI

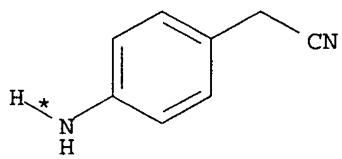


I

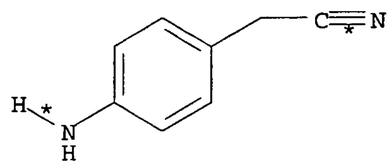
AB A series of new 6,7-disubstituted-4-(arylamino)quinoline-3-carbonitriles, e.g. I (R1 = H, Cl; R2 = PhCH2O, 1-imidazolyl, 2-furylmethoxy, etc.; R3 = Cl, CN, PhCH2O; R4 = Me, Et; R5 = Me, R6 = Me, HOCH2CH2; R5R6N = azetidinyl, piperidinyl, thiomorpholinyl, etc.) that function as irreversible inhibitors of human epidermal growth factor receptor-2



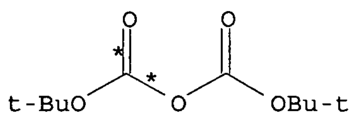
3 W



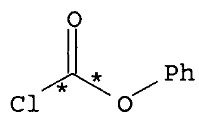
BW



BW

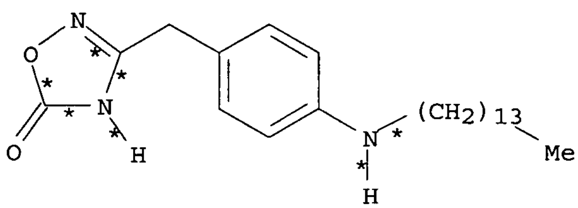


CB



DD

5 STEPS →



ED
YIELD 44%

RX(29) RCT W 112-71-0, BW **3544-25-0**
RGT BL 584-08-7 K2CO3
PRO BZ 850143-36-1, CA 850143-37-2
SOL 75-05-8 MeCN
CON 1 month, reflux

RX(30) RCT CA 850143-37-2, CB **24424-99-5**
RGT BL 584-08-7 K2CO3
PRO CC 850143-38-3
SOL 75-05-8 MeCN, 123-91-1 Dioxane
CON SUBSTAGE(1) 7 days, room temperature
SUBSTAGE(2) 14 days, reflux

RX(50) RCT CC 850143-38-3
RGT CF 5470-11-1 H2NOH-HCl, BL 584-08-7 K2CO3
PRO CY 850143-55-4
SOL 64-17-5 EtOH
CON 18 hours, reflux

RX(74) RCT CY 850143-55-4, DD 1885-14-9

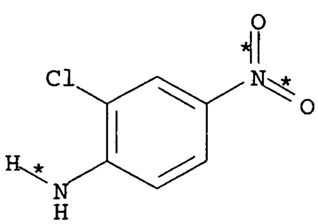
STAGE(1)
RGT E 121-44-8 Et3N
SOL 67-66-3 CHCl3
CON 1 hour, 0 deg C

(HER-2) and epidermal growth factor receptor (EGFR) kinases have been prepared These compds. demonstrated enhanced activities for inhibiting HER-2 kinase and the growth of HER-2 pos. cells compared to the EGFR kinase inhibitor I [R1 = H; R2 = F; R3 = Cl; R4 = Et; R5 = R6 = Me; (EKB-569)]. Three synthetic routes were used to prepare these compds. They were prepared mostly by acylation of 6-amino-4-(arylamino)quinoline-3-carbonitriles with unsatd. acid chlorides or by amination of 4-chloro-6-(crotonamido)quinoline-3-carbonitriles with monocyclic or bicyclic anilines. The third route was developed to prepare a key intermediate, 6-acetamido-4-chloroquinoline-3-carbonitrile, that involved a safer cyclization step. It was shown that attaching a large lipophilic group at the para position of the 4-(arylamino) ring results in improved potency for inhibiting HER-2 kinase. The importance of a basic dialkylamino group at the end of the Michael acceptor for activity, due to intramol. catalysis of the Michael addition has also been demonstrated. This, along with improved water solubility, resulted in compds. with enhanced biol. properties. The mol. modeling results consistent with the proposed mechanism of inhibition are presented. Binding studies of one compound, I [R1 = H; R2 = 2-pyridylmethoxy; R3 = Cl; R4 = Et; R5 = R6 = Me; (HKI-272)] (C-14 radiolabeled), showed that it binds irreversibly to HER-2 protein in BT474 cells. Furthermore, it demonstrated excellent oral activity, especially in HER-2 overexpressing xenografts. Compound HKI-272 was selected for further studies and is currently in phase I clin. trials for the treatment of cancer.

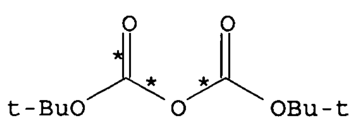
REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(290) OF 368 COMPOSED OF RX(109), RX(110), RX(111), RX(112)

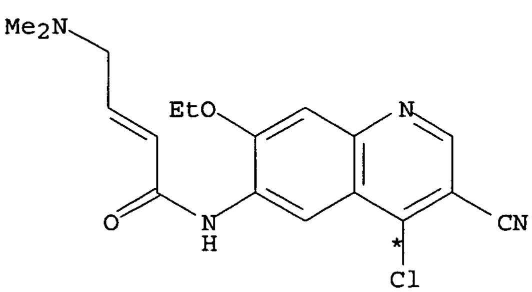
RX(290) **IK** + **IL** + GO + IR ===> **IS**



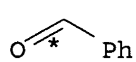
IK



IL

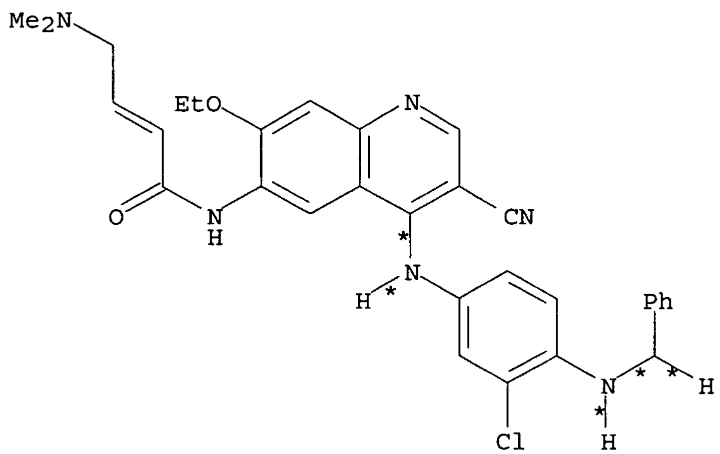


GO



IR

4 STEPS →

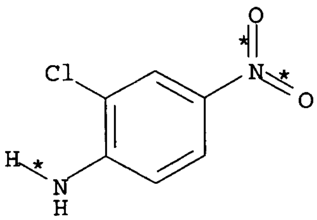


IS
YIELD 68%

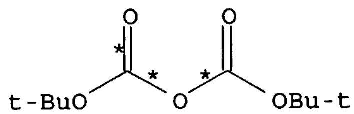
```
RX(109)   RCT  IK 121-87-9, IL 24424-99-5
          PRO  IM 252019-65-1
          CAT  1122-58-3 4-DMAP
          SOL  109-99-9 THF
          CON  SUBSTAGE(1) 0 deg C
               SUBSTAGE(2) 0 deg C -> 25 deg C
               SUBSTAGE(3) 3 hours, 25 deg C

RX(110)   RCT  IM 252019-65-1
          RGT  G 7439-89-6 Fe
          PRO  IO 252019-51-5
          SOL  67-56-1 MeOH, 64-19-7 AcOH
          CON  30 minutes, reflux

RX(111)   RCT  GO 848133-88-0, IO 252019-51-5

            STAGE(1)
               RGT  BO 628-13-7 Pyridinium chloride
               SOL  109-86-4 MeCH2CH2OH
               CON  1 hour, reflux

            STAGE(2)
               RGT  AK 584-08-7 K2CO3
               SOL  7732-18-5 Water
               CON  room temperature, pH 9

            STAGE(3)
               RGT  IQ 76-05-1 F3CCO2H
               SOL  75-09-2 CH2Cl2
               CON  SUBSTAGE(1) 0 deg C
                    SUBSTAGE(2) 0 deg C -> 25 deg C
                    SUBSTAGE(3) 15 minutes, 25 deg C

          PRO  IP 848134-31-6

RX(112)   RCT  IP 848134-31-6, IR 100-52-7
          RGT  IT 56553-60-7 Na.(AcO)3BH, I 64-19-7 AcOH
```

PRO IS **848134-32-7**
SOL 107-06-2 ClCH2CH2Cl
CON 18 hours, 25 deg C

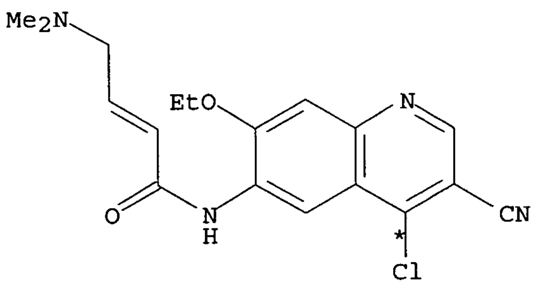
RX(291) OF 368 COMPOSED OF RX(109), RX(110), RX(111), RX(113)
RX(291) **IK** + **IL** + GO + IV ===> **IW**



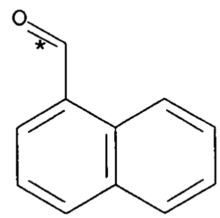
IK



IL



GO



IV

4 STEPS →

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

RX(109) RCT IK **121-87-9**, IL **24424-99-5**
PRO IM 252019-65-1
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF
CON SUBSTAGE(1) 0 deg C
SUBSTAGE(2) 0 deg C -> 25 deg C
SUBSTAGE(3) 3 hours, 25 deg C

RX(110) RCT IM 252019-65-1
RGT G 7439-89-6 Fe
PRO IO 252019-51-5
SOL 67-56-1 MeOH, 64-19-7 AcOH
CON 30 minutes, reflux

RX(111) RCT GO 848133-88-0, IO 252019-51-5

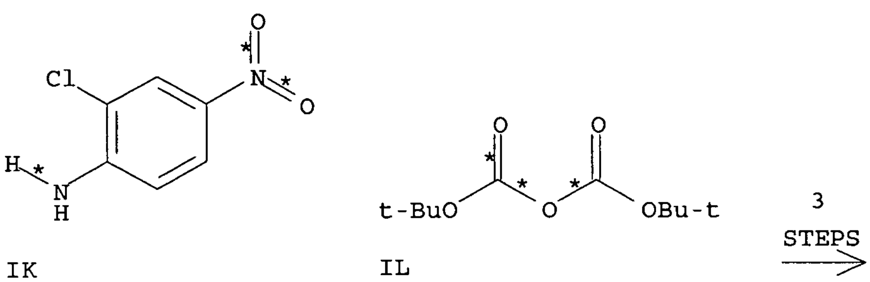
STAGE(1)
RGT BO 628-13-7 Pyridinium chloride
SOL 109-86-4 MeCH2CH2OH
CON 1 hour, reflux

```
    STAGE(2)
       RGT  AK 584-08-7 K2CO3
       SOL  7732-18-5 Water
       CON  room temperature, pH 9

    STAGE(3)
       RGT  IQ 76-05-1 F3CCO2H
       SOL  75-09-2 CH2Cl2
       CON  SUBSTAGE(1) 0 deg C
            SUBSTAGE(2) 0 deg C -> 25 deg C
            SUBSTAGE(3) 15 minutes, 25 deg C

   PRO  IP 848134-31-6

RX(113)   RCT  IP 848134-31-6, IV 66-77-3
          RGT  IT 56553-60-7 Na.(AcO)3BH, I 64-19-7 AcOH
          PRO  IW 848134-33-8
          SOL  107-06-2 ClCH2CH2Cl
          CON  18 hours, 25 deg C
```

RX(348) OF 368 COMPOSED OF REACTION SEQUENCE RX(109), RX(110), RX(111), RX(112)
AND REACTION SEQUENCE RX(85), RX(111), RX(112)

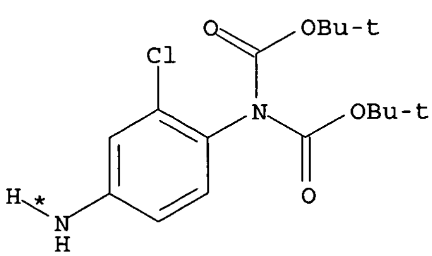
...IK + **IL** ===> IO...

...DQ + GN + **IO** + IR ===> **IS**



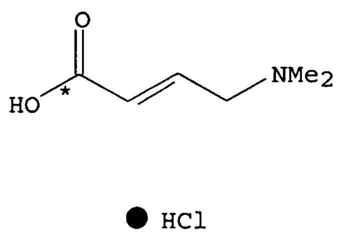
IK

IL

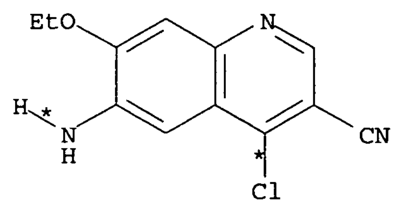


IO

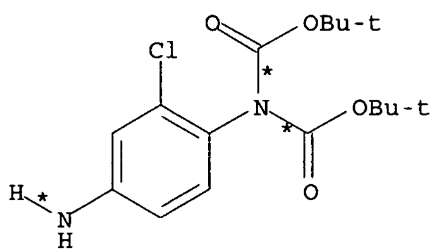
START NEXT REACTION SEQUENCE



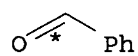
DQ



GN

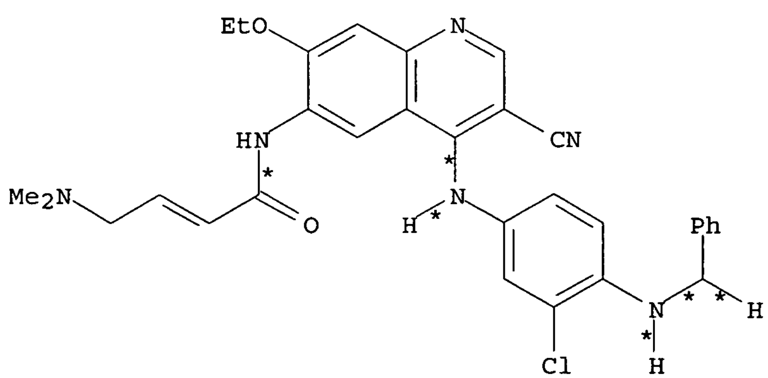


IO



IR

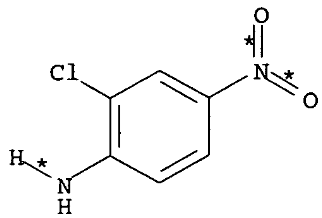
3 STEPS →



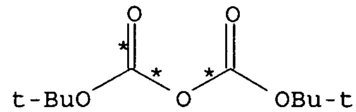
IS
YIELD 68%

```
RX(109)    RCT  IK 121-87-9, IL 24424-99-5
           PRO  IM 252019-65-1
           CAT  1122-58-3 4-DMAP
           SOL  109-99-9 THF
           CON  SUBSTAGE(1) 0 deg C
                SUBSTAGE(2) 0 deg C -> 25 deg C
                SUBSTAGE(3) 3 hours, 25 deg C

RX(110)    RCT  IM 252019-65-1
           RGT  G 7439-89-6 Fe
           PRO  IO 252019-51-5
```

```
        SOL  67-56-1 MeOH, 64-19-7 AcOH
        CON  30 minutes, reflux

RX(85)  RCT  DQ 848133-35-7

          STAGE(1)
             RGT  DT 79-37-8 (COCl)2
             CAT  68-12-2 DMF
             SOL  75-05-8 MeCN
             CON  30 minutes, 60 deg C

          STAGE(2)
             RCT  GN 848133-87-9
             SOL  872-50-4 NMEP
             CON  2.5 hours, 0 deg C

        PRO  GO 848133-88-0

RX(111) RCT  GO 848133-88-0, IO 252019-51-5

          STAGE(1)
             RGT  BO 628-13-7 Pyridinium chloride
             SOL  109-86-4 MeCH2CH2OH
             CON  1 hour, reflux

          STAGE(2)
             RGT  AK 584-08-7 K2CO3
             SOL  7732-18-5 Water
             CON  room temperature, pH 9

          STAGE(3)
             RGT  IQ 76-05-1 F3CCO2H
             SOL  75-09-2 CH2Cl2
             CON  SUBSTAGE(1) 0 deg C
                  SUBSTAGE(2) 0 deg C -> 25 deg C
                  SUBSTAGE(3) 15 minutes, 25 deg C

        PRO  IP 848134-31-6

RX(112) RCT  IP 848134-31-6, IR 100-52-7
        RGT  IT 56553-60-7 Na.(AcO)3BH, I 64-19-7 AcOH
        PRO  IS 848134-32-7
        SOL  107-06-2 ClCH2CH2Cl
        CON  18 hours, 25 deg C


RX(349) OF 368 COMPOSED OF REACTION SEQUENCE RX(109), RX(110), RX(111), RX(113)
               AND REACTION SEQUENCE RX(85), RX(111), RX(113)
...IK  +  IL  ===>  IO...
...DQ  +  GN  +  IO  +  IV  ===>  IW
```

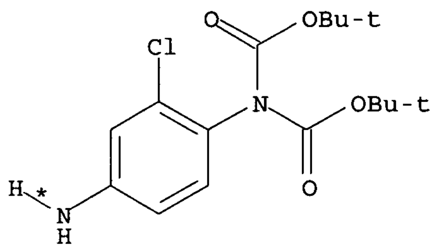


IK



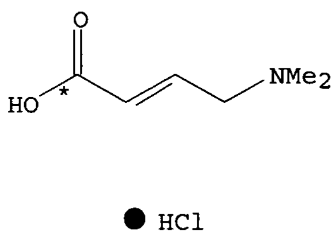
IL

3 STEPS

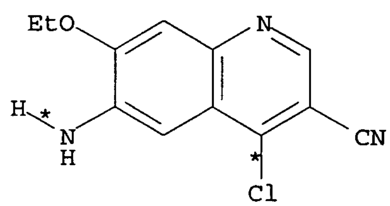


IO

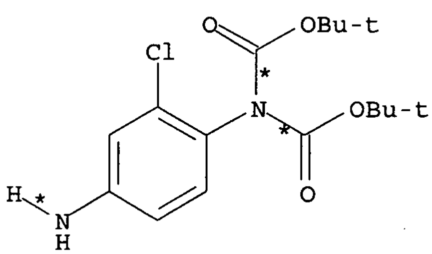
START NEXT REACTION SEQUENCE



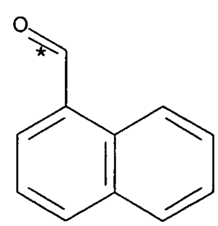
DQ



GN

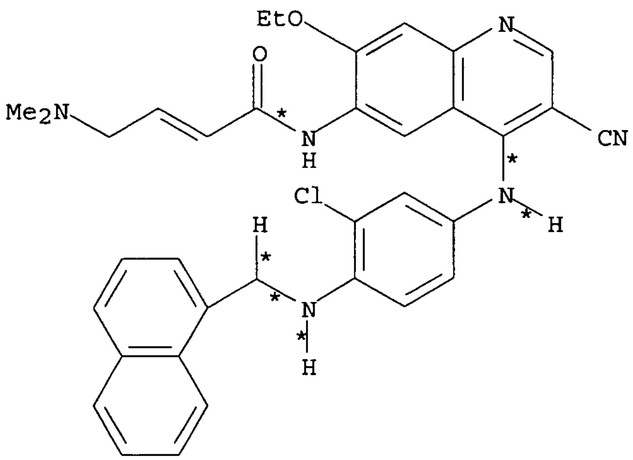


IO



IV

3 STEPS



IW
YIELD 91%

```
RX(109)    RCT  IK 121-87-9, IL 24424-99-5
           PRO  IM 252019-65-1
           CAT  1122-58-3 4-DMAP
           SOL  109-99-9 THF
           CON  SUBSTAGE(1) 0 deg C
                SUBSTAGE(2) 0 deg C -> 25 deg C
                SUBSTAGE(3) 3 hours, 25 deg C

RX(110)    RCT  IM 252019-65-1
           RGT  G 7439-89-6 Fe
           PRO  IO 252019-51-5
           SOL  67-56-1 MeOH, 64-19-7 AcOH
           CON  30 minutes, reflux

RX(85)     RCT  DQ 848133-35-7

             STAGE(1)
                RGT  DT 79-37-8 (COCl)2
                CAT  68-12-2 DMF
                SOL  75-05-8 MeCN
                CON  30 minutes, 60 deg C

             STAGE(2)
                RCT  GN 848133-87-9
                SOL  872-50-4 NMEP
                CON  2.5 hours, 0 deg C

           PRO  GO 848133-88-0

RX(111)    RCT  GO 848133-88-0, IO 252019-51-5

             STAGE(1)
                RGT  BO 628-13-7 Pyridinium chloride
                SOL  109-86-4 MeCH2CH2OH
                CON  1 hour, reflux

             STAGE(2)
```

```
            RGT  AK 584-08-7 K2CO3
            SOL  7732-18-5 Water
            CON  room temperature, pH 9

          STAGE(3)
            RGT  IQ 76-05-1 F3CCO2H
            SOL  75-09-2 CH2Cl2
            CON  SUBSTAGE(1) 0 deg C
                 SUBSTAGE(2) 0 deg C -> 25 deg C
                 SUBSTAGE(3) 15 minutes, 25 deg C

        PRO  IP 848134-31-6

RX(113) RCT  IP 848134-31-6, IV 66-77-3
        RGT  IT 56553-60-7 Na.(AcO)3BH, I 64-19-7 AcOH
        PRO  IW 848134-33-8
        SOL  107-06-2 ClCH2CH2Cl
        CON  18 hours, 25 deg C
```

L60 ANSWER 6 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 142:218805 CASREACT

TITLE: Effects of Catalyst Activation and Ligand Steric Properties on the Enantioselective Allylation of Amines and Phenoxides

AUTHOR(S): Leitner, Andreas; Shu, Chutian; Hartwig, John F.

CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 6520-8107, USA

SOURCE: Organic Letters (2005), 7(6), 1093-1096
CODEN: ORLEF7; ISSN: 1523-7060

PUBLISHER: American Chemical Society

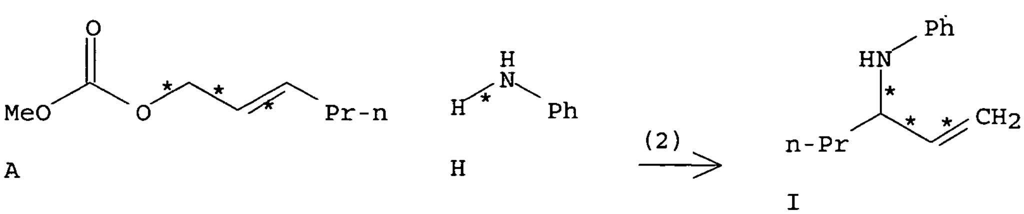
DOCUMENT TYPE: Journal

LANGUAGE: English

AB The yields, enantioselectivities, and regioselectivities of the reactions of amines and phenoxides with allylic carbonates in the presence of a metallacyclic iridium catalyst were compared. These data show that both preactivation of the catalyst and the size of the ligand affect the yield, enantioselectivity, and regioselectivity. With the activated catalyst containing a bis-naphthethylamino group, the allylic amination and etherification of a broad range of allylic carbonates occurred in high yields and with high regioselectivities and enantioselectivities.

REFERENCE COUNT: 40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(2) OF 28 **A + H ===> I**



I
YIELD 93%

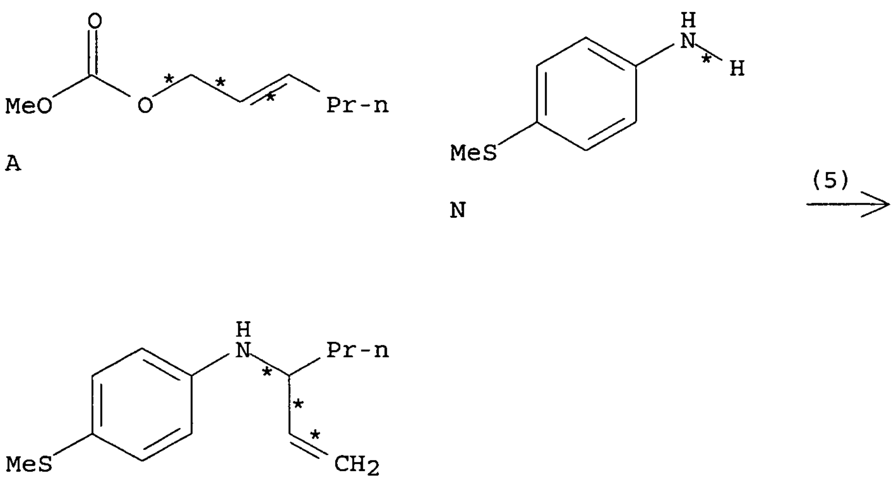
RX(2)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 107-10-8 PrNH2
SOL 109-99-9 THF
CON 20 minutes, 50 deg C

STAGE(2)
RCT A **107574-36-7**, H **62-53-3**
CON 0.5 hours, room temperature

PRO I **793726-67-7**
NTE stereoselective

RX(5) OF 28 **A + N ===> O**



O
YIELD 92%

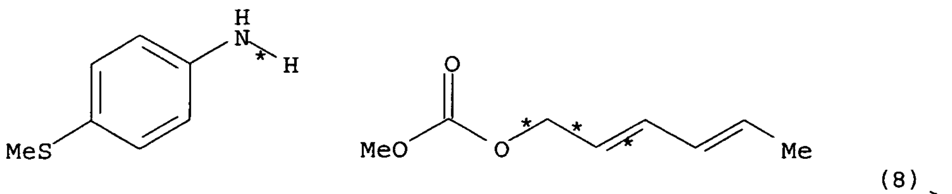
RX(5)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 107-10-8 PrNH2
SOL 109-99-9 THF
CON 20 minutes, 50 deg C

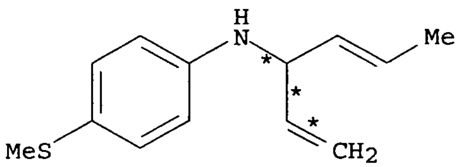
STAGE(2)
RCT A **107574-36-7**, N **104-96-1**
CON 1 hour, room temperature

PRO O **840524-20-1**
NTE stereoselective

RX(8) OF 28 **N + P ===> T**



N P



T
YIELD 93%

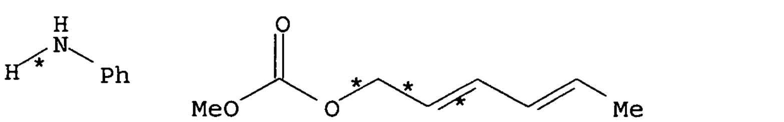
RX(8)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF

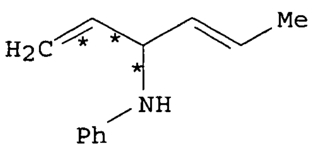
STAGE(2)
RCT N **104-96-1**, P **675608-09-0**
CON 2 hours, room temperature

PRO T **840524-23-4**
NTE stereoselective

RX(9) OF 28 **H** + **P** ===> **U**



H P



U
YIELD 81%

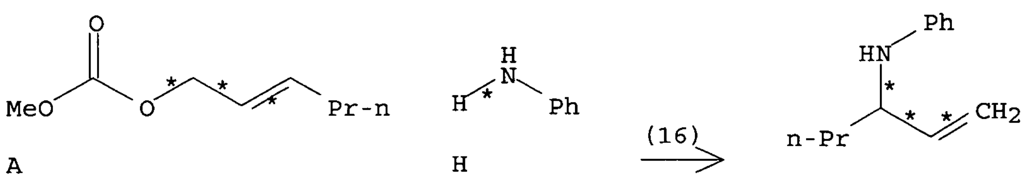
RX(9)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF

STAGE(2)
RCT H **62-53-3**, P **675608-09-0**
CON 3 hours, room temperature

PRO U **840524-24-5**
NTE stereoselective

RX(16) OF 28 **A + H ===> I**



I
YIELD 93%

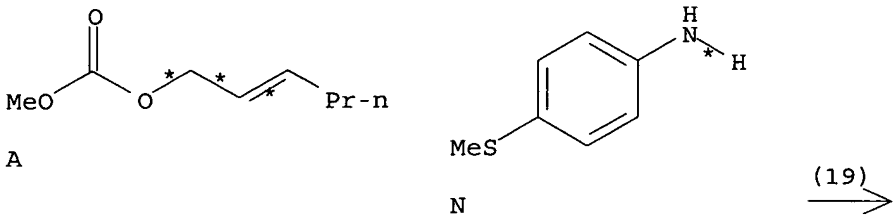
RX(16)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-, 107-10-8 PrNH2
SOL 109-99-9 THF

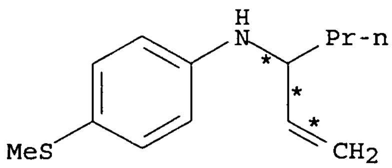
STAGE(2)
RCT A **107574-36-7**, H **62-53-3**
CON 3 hours, room temperature

PRO I **793726-67-7**
NTE stereoselective

RX(19) OF 28 **A + N ===> O**



A N



O
YIELD 94%

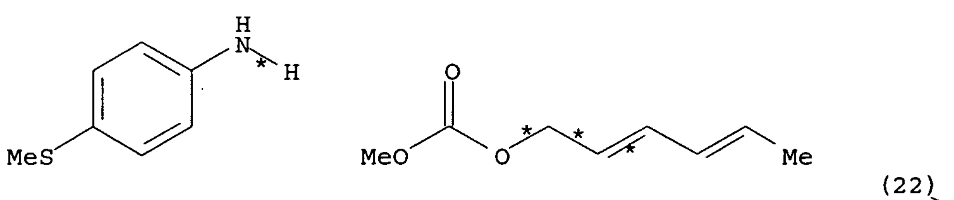
RX(19)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-, 107-10-8 PrNH2
SOL 109-99-9 THF

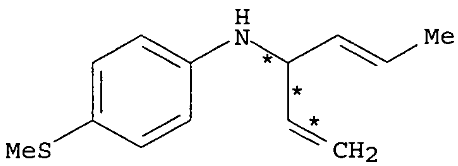
STAGE(2)
RCT A **107574-36-7**, N **104-96-1**
CON 3 hours, room temperature

PRO O **840524-20-1**
NTE stereoselective

RX(22) OF 28 **N** + **P** ===> **T**



N P



T
YIELD 73%

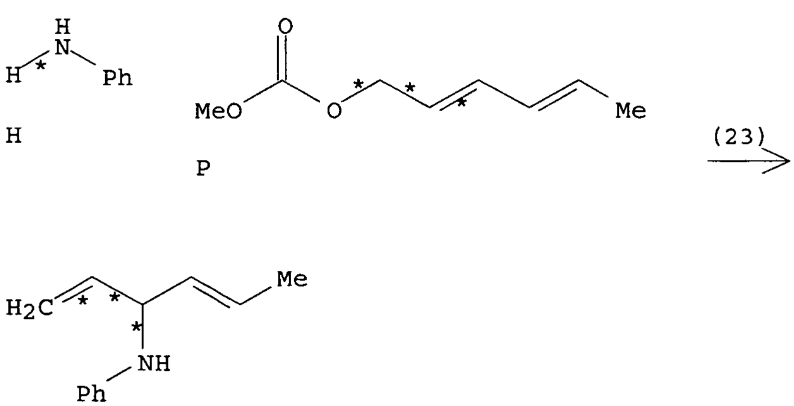
RX(22)

STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF

STAGE(2)
RCT N **104-96-1**, P **675608-09-0**
CON 18 hours, room temperature

PRO T **840524-23-4**
NTE stereoselective

RX(23) OF 28 **H** + **P** ===> **U**



U
YIELD 78%

RX(23)

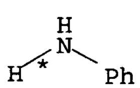
STAGE(1)
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF

STAGE(2)
RCT H **62-53-3**, P **675608-09-0**
CON 15 hours, room temperature

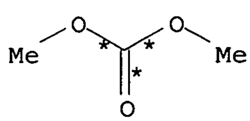
PRO U **840524-24-5**
NTE stereoselective

L60 ANSWER 7 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 142:431930 CASREACT
TITLE: Selective synthesis of N,N-dimethyl aniline derivatives using dimethyl carbonate as a methylating agent and onium salt as a catalyst
AUTHOR(S): Shivarkar, Anand B.; Gupte, Sunil P.; Chaudhari, Raghunath V.
CORPORATE SOURCE: Homogeneous Catalysis Division, National Chemical Laboratory, Pune, 411008, India
SOURCE: Journal of Molecular Catalysis A: Chemical (2005), 226(1), 49-56
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
AB N-Alkylation of anilines by di-Me carbonate (DMC) catalyzed by onium salts has been demonstrated. The work shows that a simple catalytic system consisting of onium salts in the presence of a small amount of water is extremely effective in enhancing the DMC mediated N-alkylation of anilines to dialkylated products. The effect of reaction conditions, on the synthesis of N,N-di-methylaniline (NNDMA) from aniline and DMC has been investigated. Under the optimized conditions highest yield of NNDMA obtained was 99.8%, which is the best reported for liquid phase N-alkylation of aniline using DMC. The role of water in enhancing the yield of NNDMA is explained and a reaction-networking scheme is constructed, which summarizes the chemical behind liquid phase N-alkylation of anilines by DMC. The catalyst has been shown to recycle up to five times and at the end of fifth recycle almost 98% of NNDMA yields were obtained.
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(5) OF 19 2 **A** + 3 **B** ===> C + **H**...

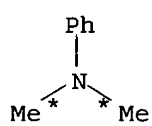


2 A

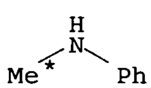


3 B

(5)



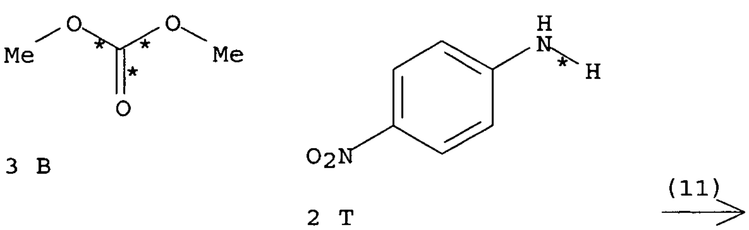
C
YIELD 15%



H
YIELD 85%

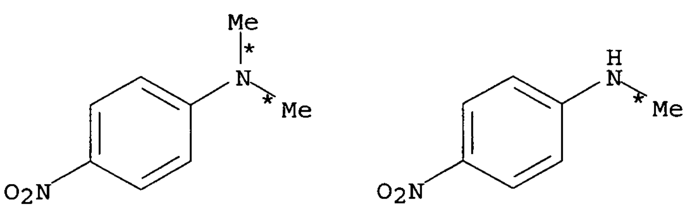
RX(5) RCT A **62-53-3**, B **616-38-6**
RGT G 7732-18-5 Water
PRO C 121-69-7, H **100-61-8**
SOL 616-38-6 Me2CO3
CON 2 hours, 433K, 34 bar
NTE Cs-Y zeolite used as catalyst, Hast 'C' high pressure Parr reactor used, green chem.-waste redn., high pressure, optimized on catalyst and temp.

RX(11) OF 19 3 **B** + 2 **T** ===> U + **V**



3 B

2 T



U
YIELD 36%

V
YIELD 27%

RX(11) RCT B **616-38-6**, T **100-01-6**
RGT G 7732-18-5 Water
PRO U 100-23-2, V **100-15-2**
CAT 71-91-0 Et4N.Br
SOL 616-38-6 Me2CO3
CON 2 hours, 443K, 34 bar
NTE Hast 'C' high pressure Parr reactor used, green chem.-waste redn., high pressure

L60 ANSWER 8 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 140:287536 CASREACT
TITLE: Preparation of phosphoramidite iridium complexes as catalysts for enantioselective amination and etherification reaction
INVENTOR(S): Hartwig, John F.; Shu, Chutian; Ohmura, Toshimichi; Kiener, Christoph; Lopez, Fernando Garcia
PATENT ASSIGNEE(S): Yale University, USA
SOURCE: PCT Int. Appl., 100 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004024684	A2	20040325	WO 2003-US28718	20030912
WO 2004024684	A3	20040513		

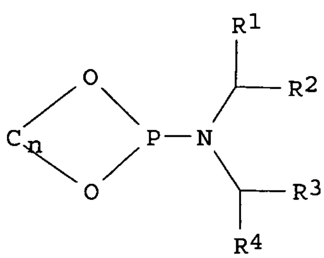
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

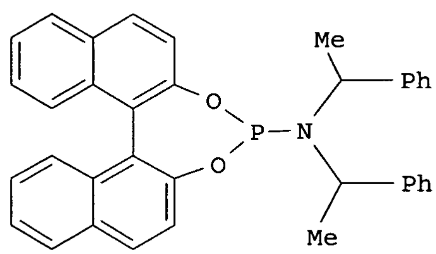
AU 2003272349	A1	20040430	AU 2003-272349	20030912
PRIORITY APPLN. INFO.:			US 2002-410407P	20020913
			US 2003-445154P	20030205
			WO 2003-US28718	20030912

OTHER SOURCE(S): MARPAT 140:287536

GI



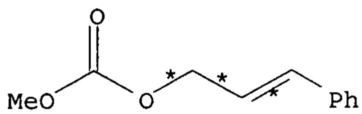
I



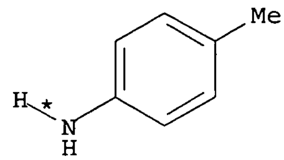
II

AB The present invention is directed to a catalyst composition, comprising: (1) a catalyst precursor having the general structure MSXn (M = a transition metal selected from the group consisting of iridium, molybdenum, and tungsten; S = a coordinating ligand; X = a counterion; n = 0-5); and (2) a phosphoramidite ligand I (O-Cn-O = aliphatic or aromatic diolate and wherein R1, R2, R3, and R4 are selected from the group consisting of substituted or unsubstituted aryl groups, substituted or unsubstituted heteroaryl groups, substituted or unsubstituted aliphatic groups, and combinations thereof, with the proviso that at least one of R1, R2, R3, or R4 must be a substituted or unsubstituted aryl or heteroaryl group). The present invention is also directed to activated catalysts made from the above catalyst composition, as well as methods of allylic amination and etherification using the above catalysts. Thus, [Ir(cod)Cl]2/phosphoramidite ligand II catalyzed enantioselective allylic amination of cinnamyl Me carbonate with benzylamine at room temperature in 10h gave 84% N-(1-phenyl-2-propenyl)benzylamine. The crystal structures of two phosphoramidite iridium complexes were determined

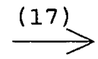
RX(17) OF 54 **N** + **AM** ===> **AN**

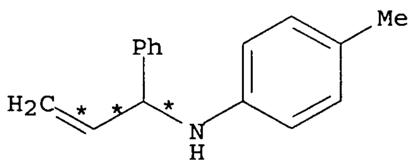


N



AM

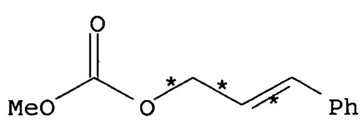




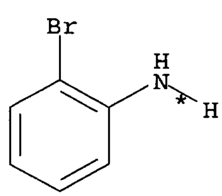
AN
YIELD 76%

RX(17)	RCT	N **85217-69-2**, AM **106-49-0**
	RGT	AO 280-57-9 Triethylenediamine
	PRO	AN **675608-10-3**
	CAT	676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
	SOL	109-99-9 THF
	CON	6 hours, room temperature
	NTE	stereoselective

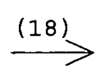
RX(18) OF 54 **N** + **AQ** ===> **AR**

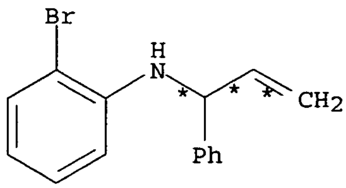


N



AQ

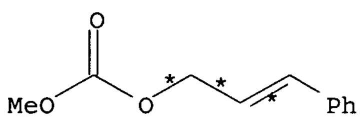




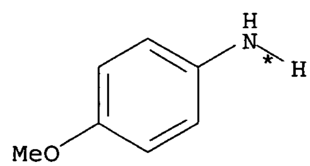
AR
YIELD 66%

RX(18) RCT N **85217-69-2**, AQ **615-36-1**
RGT AO 280-57-9 Triethylenediamine
PRO AR **675608-11-4**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 16 hours, room temperature
NTE stereoselective

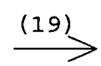
RX(19) OF 54 **N + AS ===> AT**

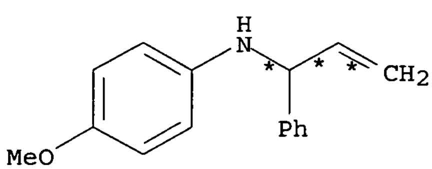


N



AS



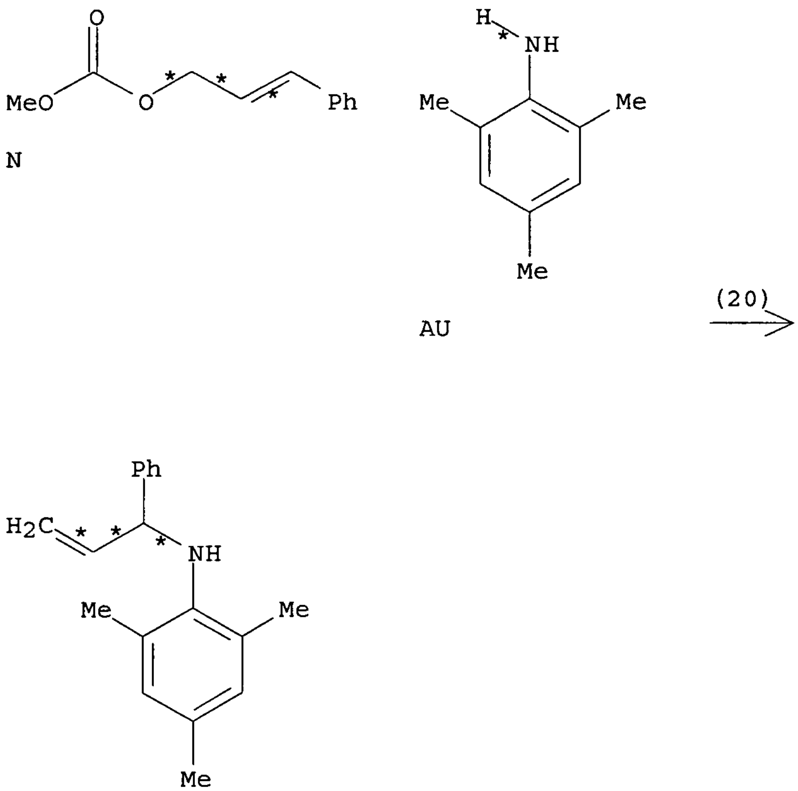


AT
YIELD 91%

RX(19) RCT N **85217-69-2**, AS **104-94-9**
RGT AO 280-57-9 Triethylenediamine
PRO AT **104937-84-0**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF

CON 16 hours, room temperature
NTE stereoselective

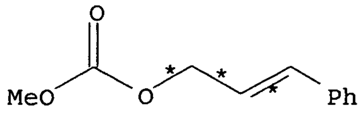
RX(20) OF 54 **N** + **AU** ===> **AV**



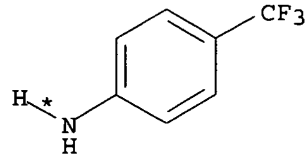
AV
YIELD 85%

RX(20) RCT N **85217-69-2**, AU **88-05-1**
RGT AO 280-57-9 Triethylenediamine
PRO AV **675608-12-5**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 12 hours, room temperature
NTE stereoselective

RX(21) OF 54 **N** + **AW** ===> **AX**

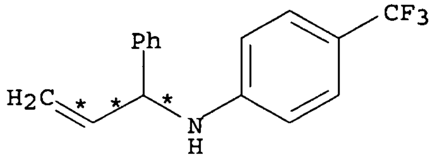


N



AW

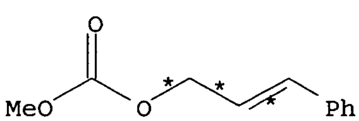
(21) →



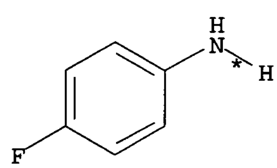
AX
YIELD 72%

RX(21) RCT N **85217-69-2**, AW **455-14-1**
RGT AO 280-57-9 Triethylenediamine
PRO AX **675608-13-6**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 12 hours, room temperature
NTE stereoselective

RX(22) OF 54 **N** + **AY** ===> **AZ**

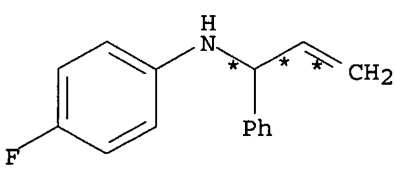


N



AY

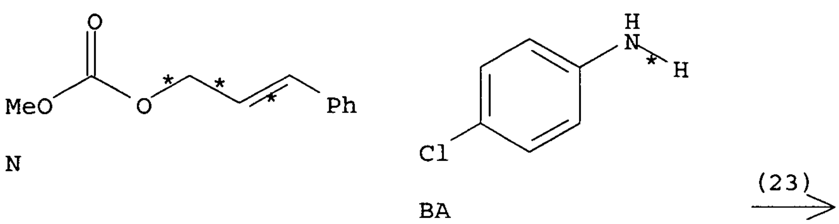
(22) →



AZ
YIELD 96%

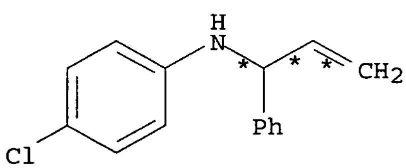
RX(22) RCT N **85217-69-2**, AY **371-40-4**
RGT AO 280-57-9 Triethylenediamine
PRO AZ **675608-14-7**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 12 hours, room temperature
NTE stereoselective

RX(23) OF 54 **N** + **BA** ===> **BB**



N

BA



BB
YIELD 95%

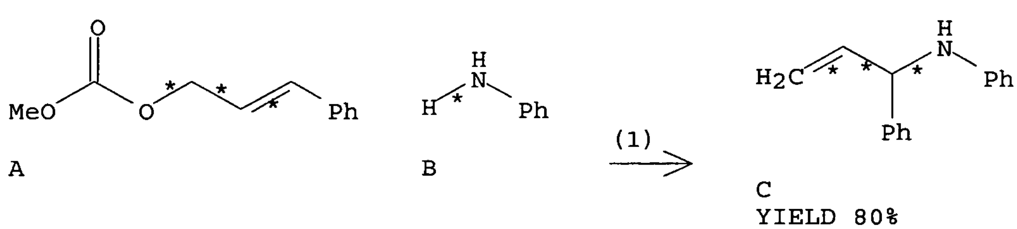
RX(23) RCT N **85217-69-2**, BA **106-47-8**
RGT AO 280-57-9 Triethylenediamine
PRO BB **345642-59-3**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 16 hours, room temperature
NTE stereoselective

RX(24) OF 54 **N** + **BC** ===> **BD**

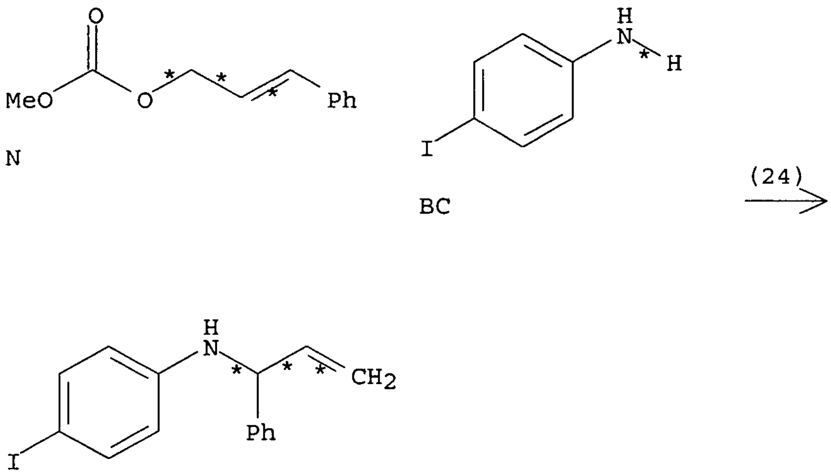
RX(25) RCT N **85217-69-2**, BE **536-90-3**
RGT AO 280-57-9 Triethylenediamine
PRO BF **675608-16-9**
CAT 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 12 hours, room temperature
NTE stereoselective

L60 ANSWER 9 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 141:423999 CASREACT
TITLE: Synthesis of allylic amines: Enantioselective allylation of aromatic amines after in situ generation of an activated cyclometalated iridium catalyst
AUTHOR(S): Shu, Chutian; Leitner, Andreas; Hartwig, John F.
CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 06520-8107, USA
SOURCE: Angewandte Chemie, International Edition (2004), 43(36), 4797-4800
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Highly regio- and enantioselective allylation of aromatic amines is observed when a cyclometalated Ir-phosphoramidite complex is generated in situ. The active catalyst can be formed from [{Ir(cod)Cl}2] and ligand L with a volatile alkylamine prior to addition of the reagents or upon use of a tertiary amine additive. E.g., a cyclometalated Ir-phosphoramidite complex catalyzed the allylation of PhNH2 by carbonate (E)-PhCH:CHCH2OC(O)OMe to give 80% (+)-PhCH(NHPh)CH:CH2.
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 27 **A + B ===> C**



RX(1) RCT A **85217-69-2**, B **62-53-3**
PRO C **793726-44-0**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective



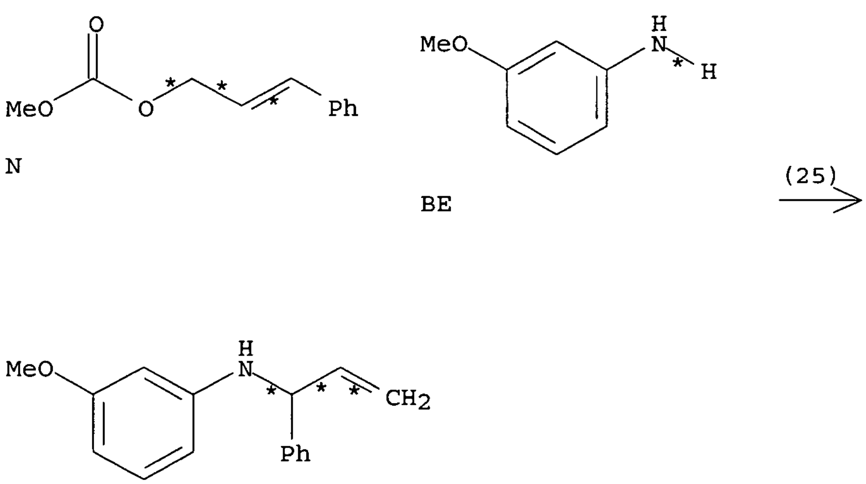
N

BC

BD
YIELD 92%

```
RX(24)      RCT  N 85217-69-2, BC 540-37-4
            RGT  AO 280-57-9 Triethylenediamine
            PRO  BD 675608-15-8
            CAT  676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-
                 amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-,
                 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-
                 cyclooctadiene]di-
            SOL  109-99-9 THF
            CON  12 hours, room temperature
            NTE  stereoselective
```

RX(25) OF 54 **N** + **BE** ===> **BF**

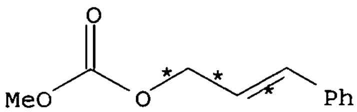


N

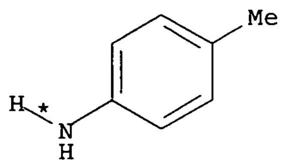
BE

BF
YIELD 82%

RX(2) OF 27 **A + G ===> H**

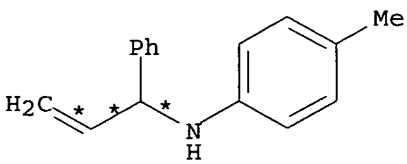


A



G

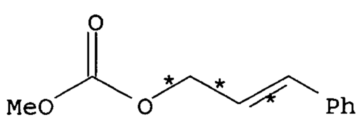
$\xrightarrow{(2)}$



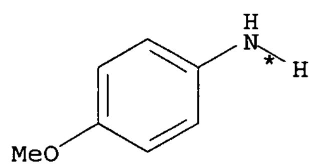
H
YIELD 76%

RX(2) RCT A **85217-69-2**, G **106-49-0**
PRO H **793726-45-1**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 6 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(3) OF 27 **A + I ===> J**

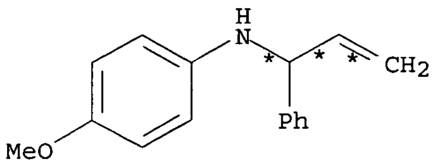


A



I

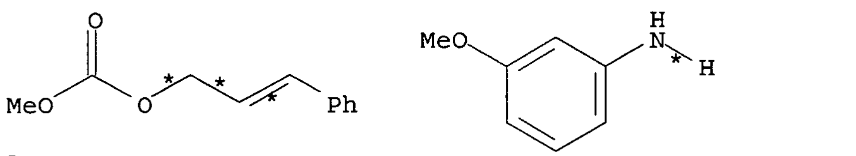
$\xrightarrow{(3)}$



J
YIELD 91%

RX(3) RCT A **85217-69-2**, I **104-94-9**
PRO J **793726-47-3**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 4 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

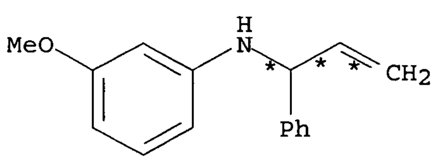
RX(4) OF 27 **A + K ===> L**



A

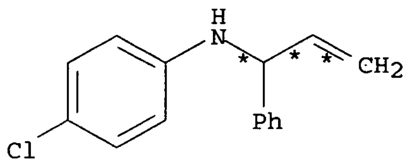
K

(4)



L
YIELD 82%

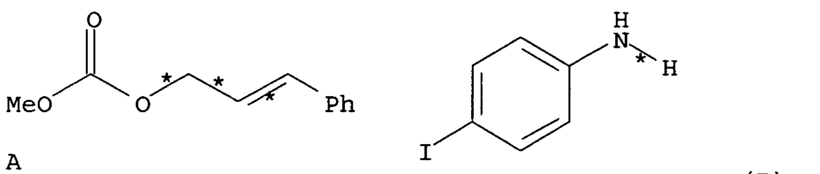
RX(4) RCT A **85217-69-2**, K **536-90-3**
PRO L **793726-48-4**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 4 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective



P
YIELD 89%

RX(6) RCT A **85217-69-2**, O **106-47-8**
PRO P **793726-52-0**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

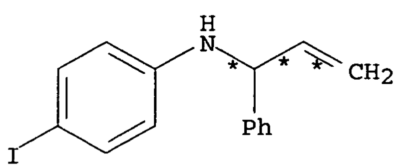
RX(7) OF 27 **A + Q ===> R**



A

Q

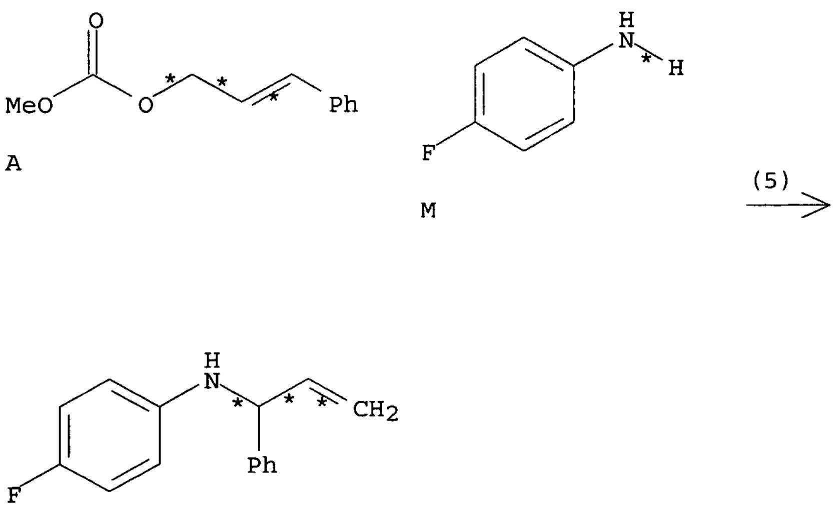
(7)



R
YIELD 92%

RX(7) RCT A **85217-69-2**, Q **540-37-4**
PRO R **793726-53-1**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

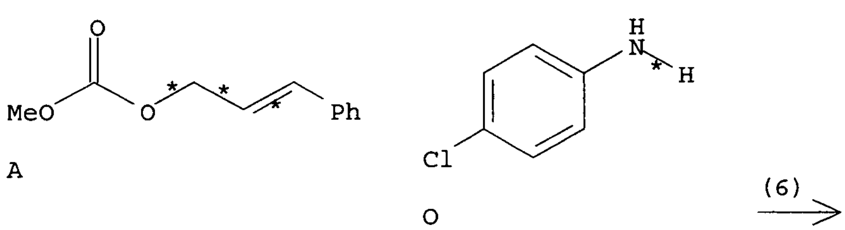
RX(5) OF 27 **A** + **M** ===> **N**



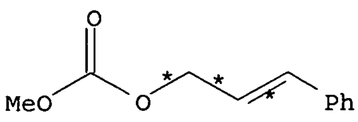
N
YIELD 90%

RX(5) RCT A **85217-69-2**, M **371-40-4**
PRO N **793726-51-9**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

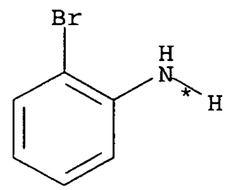
RX(6) OF 27 **A** + **O** ===> **P**



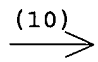
RX(10) OF 27 **A + W ===> X**

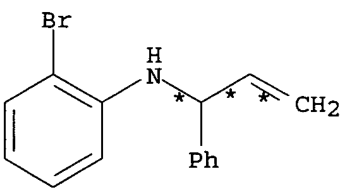


A



W

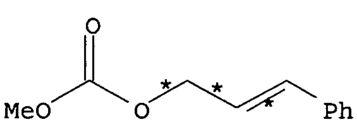




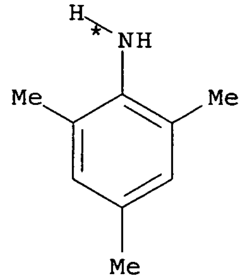
X
YIELD 66%

RX(10) RCT A **85217-69-2**, W **615-36-1**
PRO X **793726-57-5**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 16 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

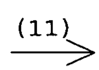
RX(11) OF 27 **A + Y ===> Z**

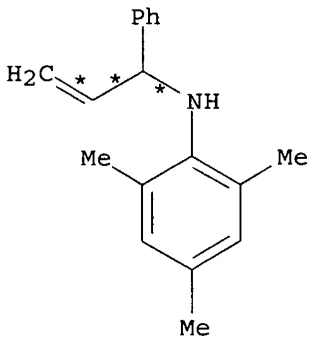


A



Y

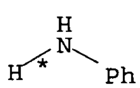




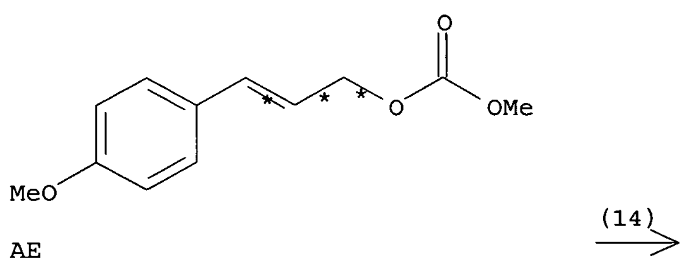
Z
YIELD 82%

```
RX(11)     RCT  A 85217-69-2, Y 88-05-1
           PRO  Z 793726-58-6
           CAT  12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-
                cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-
                f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-
                naphthalenyl)ethyl]-, (11bR)-
           SOL  109-99-9 THF
           CON  2 hours, room temperature
           NTE  in-situ generated catalyst, regioselective, stereoselective
```

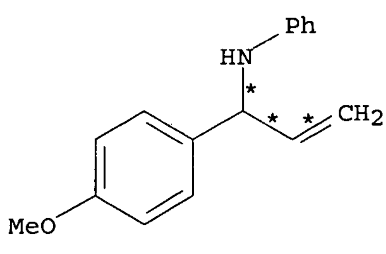
RX(14) OF 27 **B** + **AE** ===> **AF**



B



AE

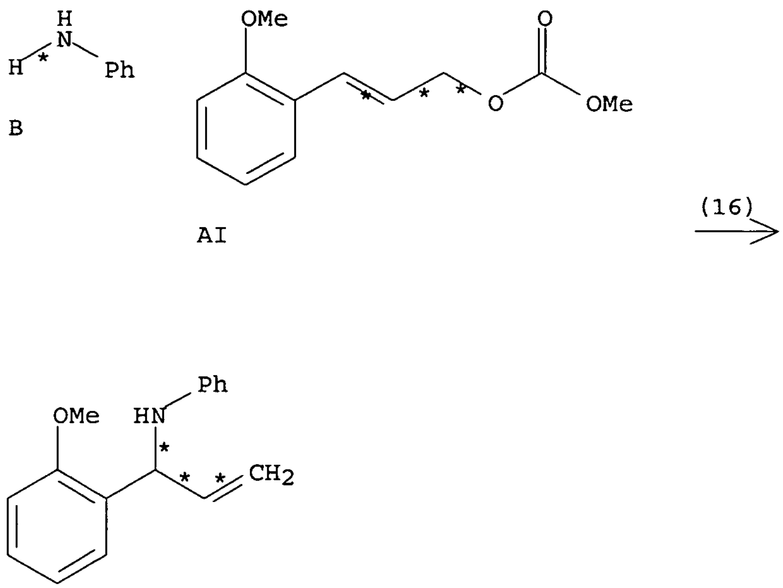


AF
YIELD 88%

```
RX(14)     RCT  B 62-53-3, AE 496789-06-1
           PRO  AF 793726-64-4
```

CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

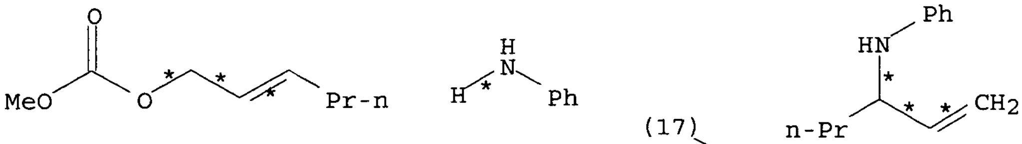
RX(16) OF 27 **B** + **AI** ===> **AJ**



AJ
YIELD 91%

RX(16) RCT B **62-53-3**, AI **512789-12-7**
PRO AJ **793726-66-6**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 10 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

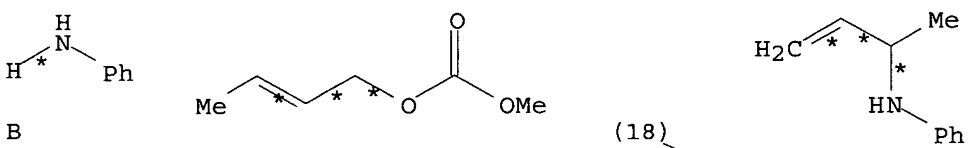
RX(17) OF 27 **AK** + **B** ===> **AL**



AK B AL
YIELD 93%

RX(17) RCT AK **107574-36-7**, B **62-53-3**
PRO AL **793726-67-7**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 2.5 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

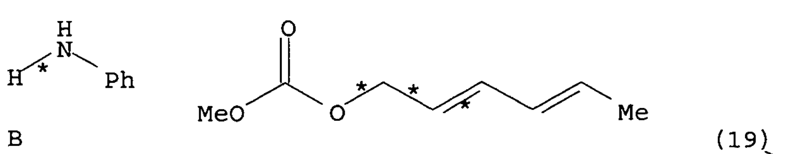
RX(18) OF 27 **B + AM ===> AN**



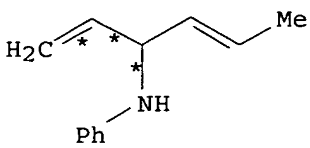
B AM AN
YIELD 72%

RX(18) RCT B **62-53-3**, AM **103185-13-3**
PRO AN **70094-92-7**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 3 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(19) OF 27 **B + AO ===> AP**



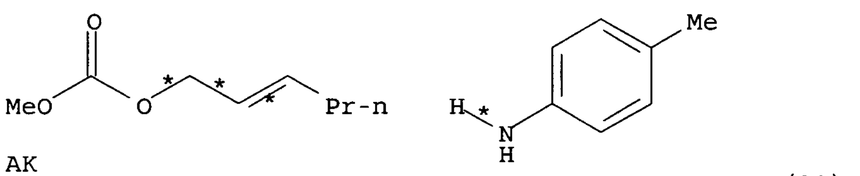
B AO



AP
YIELD 87%

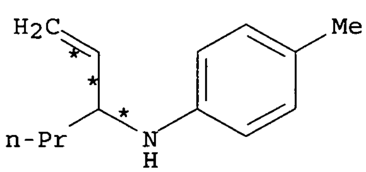
RX(19) RCT B **62-53-3**, AO **675608-09-0**
PRO AP **793726-69-9**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-
SOL 109-99-9 THF
CON 3 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(20) OF 27 **AK** + **G** ===> **AQ**



AK

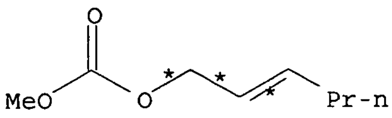
G



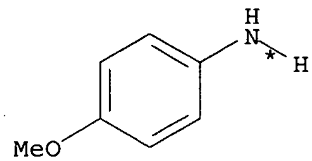
AQ
YIELD 94%

RX(20) RCT AK **107574-36-7**, G **106-49-0**
PRO AQ **793726-46-2**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-
SOL 109-99-9 THF
CON 2 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

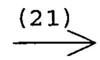
RX(21) OF 27 **AK** + **I** ===> **AS**

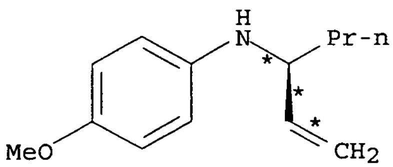


AK



I

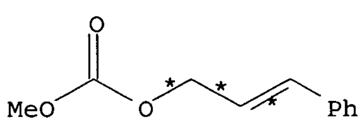




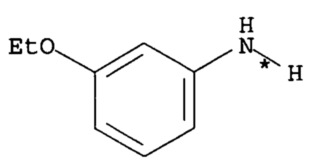
AS
YIELD 95%

RX(21) RCT AK **107574-36-7**, I **104-94-9**
PRO AS **562870-91-1**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-
SOL 109-99-9 THF
CON 2 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

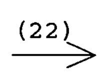
RX(22) OF 27 **A** + **AT** ===> **AU**

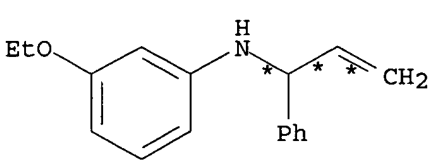


A



AT

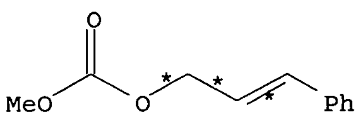




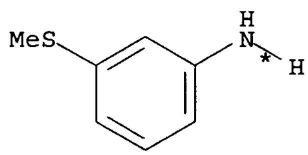
AU
YIELD 86%

RX(22) RCT A **85217-69-2**, AT **621-33-0**
PRO AU **793726-50-8**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-
SOL 109-99-9 THF
CON 2.5 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(23) OF 27 **A + AV ===> AW**

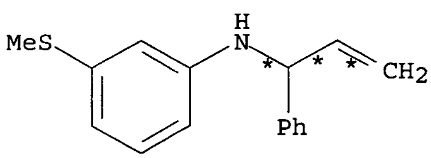


A



AV

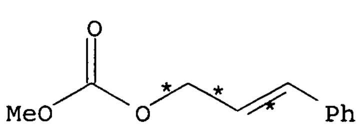
(23) →



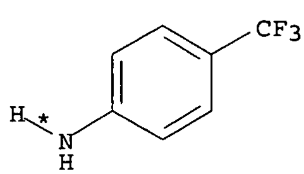
AW
YIELD 85%

RX(23) RCT A **85217-69-2**, AV **1783-81-9**
PRO AW **793726-59-7**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 415918-91-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-phenylethyl]-, (11bR)-
SOL 109-99-9 THF
CON 2.5 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(24) OF 27 **A + AX ===> AY**

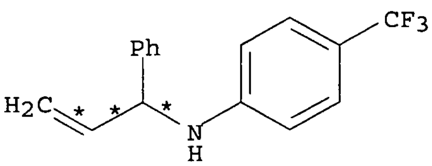


A



AX

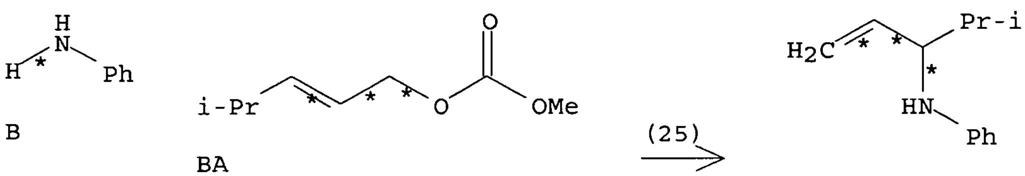
(24) →



AY
YIELD 72%

RX(24) RCT A **85217-69-2**, AX **455-14-1**
PRO AY **793726-56-4**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF
CON 16 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

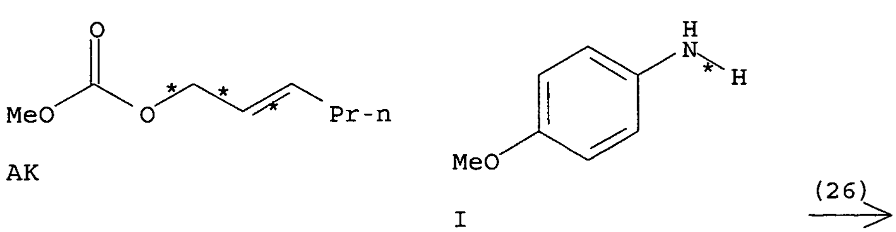
RX(25) OF 27 **B + BA ===> BB**



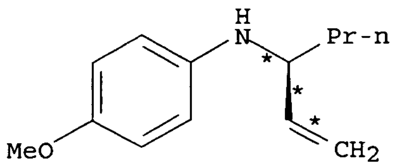
B BA BB
YIELD 83%

RX(25) RCT B **62-53-3**, BA **675608-08-9**
PRO BB **793726-70-2**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF
CON 16 hours, room temperature
NTE in-situ generated catalyst, regioselective, stereoselective

RX(26) OF 27 **AK + I ===> AS**



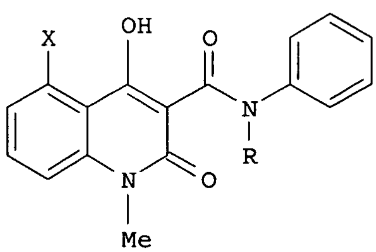
AK I



AS
YIELD 85%

RX(26) RCT AK **107574-36-7**, I **104-94-9**
PRO AS **562870-91-1**
CAT 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-, 676127-12-1 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine, N,N-bis[(1R)-1-(1-naphthalenyl)ethyl]-, (11bR)-, 280-57-9 Triethylenediamine
SOL 109-99-9 THF
CON 50 deg C
NTE in-situ generated catalyst, regioselective, stereoselective

L60 ANSWER 10 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 140:287248 CASREACT
TITLE: Synthesis and Biological Evaluation of New 1,2-Dihydro-4-hydroxy-2-oxo-3-quinolinecarboxamides for Treatment of Autoimmune Disorders: Structure-Activity Relationship
AUTHOR(S): Joensson, Stig; Andersson, Gunnar; Fex, Tomas; Fristedt, Tomas; Hedlund, Gunnar; Jansson, Karl; Abramo, Lisbeth; Fritzson, Ingela; Pekarski, Olga; Runstroem, Anna; Sandin, Helena; Thuvesson, Ingela; Bjoerk, Anders
CORPORATE SOURCE: Active Biotech Research AB, Lund, SE-220 07, Swed.
SOURCE: Journal of Medicinal Chemistry (2004), 47(8), 2075-2088
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



I

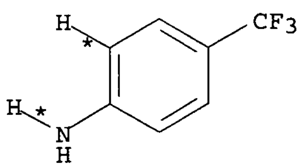
AB Roquinimex-related 3-quinolinecarboxamide derivs., e.g. I (R = Et, X = Cl), were prepared and evaluated for treatment of autoimmune disorders. The

compds. were tested in mice for their inhibitory effects on disease development in the acute exptl. autoimmune encephalomyelitis model and selected compds. in the beagle dog for induction of proinflammatory reaction. Structure-activity relationships are discussed. Compound I (R = Et, X = Cl), laquinimod, showed improved potency and superior toxicol. profile compared to the lead compound roquinimex (I; R = Me, X = H) and was selected for clin. studies (currently in phase II).

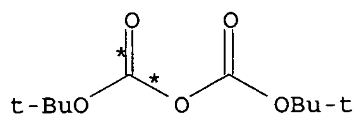
REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(173) OF 182 COMPOSED OF RX(94), RX(95)

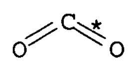
RX(173) **BR** + **BC** + BA + C + AH ===> **FW**



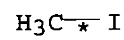
BR



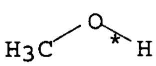
BC



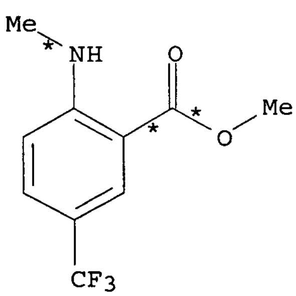
BA



C



AH



FW

RX(94) RCT BR **455-14-1**, BC **24424-99-5**

STAGE(1)
SOL 109-99-9 THF
CON 18 hours, reflux

STAGE(2)
RGT BD 594-19-4 t-BuLi
SOL 109-99-9 THF, 110-54-3 Hexane
CON 2.5 hours, -40 deg C

STAGE(3)

```
            RCT  BA 124-38-9
            CON  SUBSTAGE(1) -40 deg C
                 SUBSTAGE(2) -40 deg C -> 0 deg C

         STAGE(4)
            RGT  G 7732-18-5 Water
            CON  0 deg C

        PRO  FV 141940-29-6
        NTE  regioselective

RX(95)  RCT  FV 141940-29-6

         STAGE(1)
            RGT  F 7646-69-7 NaH
            SOL  68-12-2 DMF
            CON  1 hour, 0 deg C

         STAGE(2)
            RCT  C 74-88-4
            CON  SUBSTAGE(1) 0 deg C
                 SUBSTAGE(2) overnight, room temperature

         STAGE(3)
            RGT  G 7732-18-5 Water

         STAGE(4)
            RCT  AH 67-56-1
            RGT  AD 7647-01-0 HCl
            SOL  7732-18-5 Water, 67-56-1 MeOH
            CON  overnight, room temperature

         STAGE(5)
            RGT  BE 1310-73-2 NaOH
            SOL  7732-18-5 Water

        PRO  FW **675575-41-4**
```

L60 ANSWER 11 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 140:289172 CASREACT

TITLE: Carbamate Synthesis on Pd/C Catalysts: Gas-Solid versus Slurry Processes

AUTHOR(S): Toochinda, Pisanu; Chuang, Steven S. C.

CORPORATE SOURCE: Department of Chemical Engineering, The University of Akron, Akron, OH, 44325-3906, USA

SOURCE: Industrial & Engineering Chemistry Research (2004), 43(5), 1192-1199

CODEN: IECRED; ISSN: 0888-5885

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

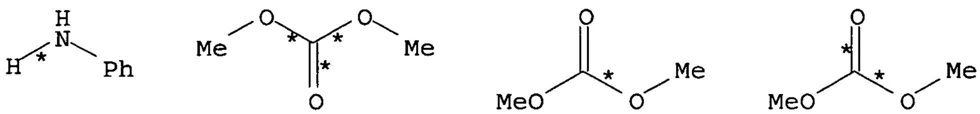
LANGUAGE: English

AB Carbamate synthesis has been studied over NaI-Pd/C in slurry, gas-solid, and tubular reactors at 373-438 K and 0.41-7.61 MPa. The gas-solid carbamate synthesis process in which the CO, O2, methanol, and aniline reactants are present in the gas phase and the catalyst is in solid form occurs at a significantly higher rate than the slurry-phase synthesis in which CO and O2 dissolve in the liquid methanol/aniline mixture The high rate of the gas-solid carbamate synthesis compared with that of the slurry-phase synthesis can be attributed to (i) the intimate contact

between the NaI promoter and the Pd on the carbon surface, (ii) the absence of solubility limitations in the gas-solid synthesis, and (iii) the slowing of the sintering of the Pd particles. Reaction pathway studies show that the direct oxidative carbonylation of aniline with methanol is the most effective pathway for carbamate synthesis. A low-cost, environmentally benign carbamate synthesis for the replacement of the isocyanate synthesis from phosgene/amine can be developed by coupling the high rate of the gas-solid synthesis with its intrinsic advantage of ease of catalyst recovery.

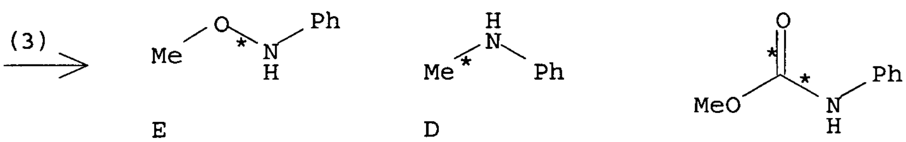
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(3) OF 5 3 **J** + 3 **L** ===> E + **D** + C



3 J L L L

(3)



E
YIELD 39%

D
YIELD 32%

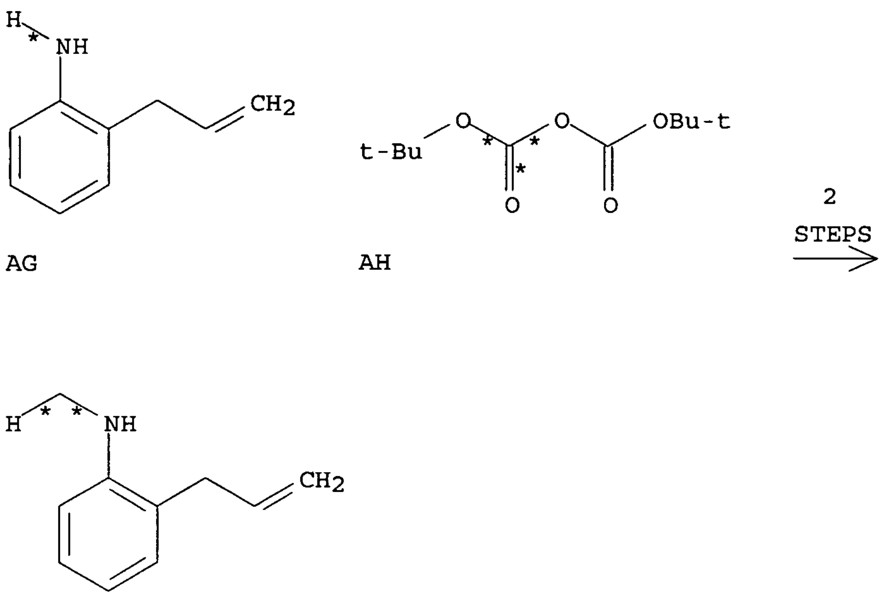
C
YIELD 1%

RX(3) RCT J **62-53-3**, L **616-38-6**
PRO E 32654-23-2, D **100-61-8**, C 2603-10-3
CAT 7681-82-5 NaI, 7440-05-3 Pd, 7440-44-0 Carbon
CON 480 minutes, 453K, 2.42 MPa
NTE thermal, Fixed bed reactor used, alternative reaction conditions (slurry method) gave lower yield, alternative reaction conditions shown, high pressure

L60 ANSWER 12 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 141:71421 CASREACT
TITLE: Zirconium catalyzed enantioselective hydroamination/cyclization
AUTHOR(S): Knight, Paul D.; Munslow, Ian; O'Shaughnessy, Paul N.; Scott, Peter
CORPORATE SOURCE: Department of Chemistry, University of Warwick, Coventry, UK
SOURCE: Chemical Communications (Cambridge, United Kingdom) (2004), (7), 894-895
CODEN: CHCOFS; ISSN: 1359-7345
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A chiral zirconium alkyl cation catalyzes the cyclization of certain aminoalkenes with enantioselectivity up to 82%, the highest thus far observed

for such a process.
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(23) OF 26 COMPOSED OF RX(12), RX(5)
RX(23) **AG** + **AH** ===> **S**



S
YIELD 59%

RX(12) RCT AG **32704-22-6**, AH **24424-99-5**
RGT AI 121-44-8 Et3N
PRO R 309974-70-7
SOL 109-99-9 THF
CON 1 day, reflux

RX(5) RCT R 309974-70-7

STAGE(1)
RGT H 16853-85-3 LiAlH4
SOL 109-99-9 THF
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) 1 day, reflux
SUBSTAGE(3) reflux -> 0 deg C

STAGE(2)
RGT K 7732-18-5 Water
CON 0 deg C

PRO S **41652-73-7**

L60 ANSWER 13 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 141:277290 CASREACT

TITLE: Selective N,N-dimethylation of primary aromatic amines with dimethyl carbonate in the presence of diphenylammonium triflate

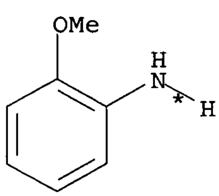
AUTHOR(S): Shen, Zhen Lu; Jiang, Xuan Zhen
CORPORATE SOURCE: Department of Chemistry, Zhejiang University, Hangzhou, Zhejiang, 310027, Peop. Rep. China

SOURCE: Journal of Molecular Catalysis A: Chemical (2004), 213(2), 193-198
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English

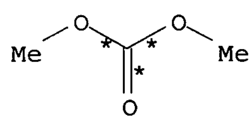
AB A facile synthesis of N,N-dimethylanilines from primary aromatic amines and di-Me carbonate has been achieved for the first time in the presence of diphenylammonium triflate. N,N-Dimethylanilines were selectively obtained in high yields. All the reaction parameters, viz. reaction temperature, time, molar ratio of reactants, and catalyst amount, were examined in the reaction of aniline and di-Me carbonate. The conversion of aniline and the selectivity to N,N-dimethylaniline were 99.9 and 99.6%, resp., at 180° in 160 min.

REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(6) OF 11 2 **M** + 3 **A** ===> N + **O**

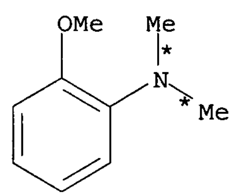


2 M

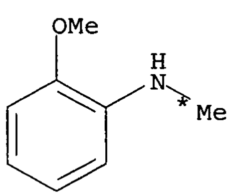


3 A

(6) ⟶



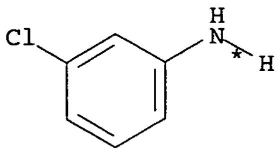
N
YIELD 97%



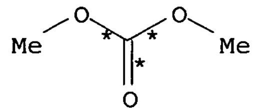
O

RX(6) RCT M **90-04-0**, A **616-38-6**
PRO N 700-75-4, O **10541-78-3**
CAT 164411-06-7 Methanesulfonic acid, trifluoro-, compd. with N-phenylbenzenamine (1:1)
CON SUBSTAGE(1) 230 minutes, 180 deg C
SUBSTAGE(2) 180 deg C -> room temperature
NTE chemoselective, thermal, autoclave used, green chem.-waste redn., no solvent, other product(s) also detected

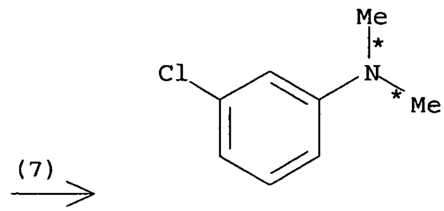
RX(7) OF 11 2 **P** + 3 **A** ===> Q + **R**



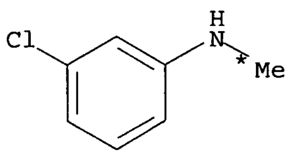
2 P



3 A



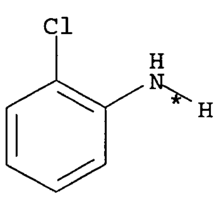
Q
YIELD 95%



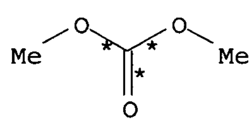
R

RX(7) RCT P **108-42-9**, A **616-38-6**
PRO Q 6848-13-1, R **7006-52-2**
CAT 164411-06-7 Methanesulfonic acid, trifluoro-, compd. with N-phenylbenzenamine (1:1)
CON SUBSTAGE(1) 300 minutes, 180 deg C
SUBSTAGE(2) 180 deg C -> room temperature
NTE chemoselective, thermal, autoclave used, green chem.-waste redn., no solvent, other product(s) also detected

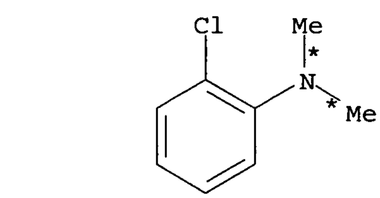
RX(9) OF 11 2 **U** + 3 **A** ===> V + **W**



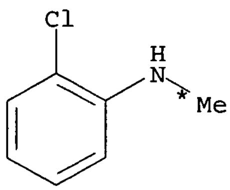
2 U



3 A



V
YIELD 42%



W

RX(9) RCT U **95-51-2**, A **616-38-6**
PRO V 698-01-1, W **932-32-1**
CAT 164411-06-7 Methanesulfonic acid, trifluoro-, compd. with N-phenylbenzenamine (1:1)
CON SUBSTAGE(1) 300 minutes, 180 deg C
SUBSTAGE(2) 180 deg C -> room temperature
NTE chemoselective, thermal, autoclave used, green chem.-waste redn., no solvent, other product(s) also detected

L60 ANSWER 14 OF 44 CASREACT

ACCESSION NUMBER: 142:55972 CASREACT

TITLE: Carbomethoxylating reactivity of methyl phenyl carbonate toward aromatic amines in the presence of group 3 metal (Sc, La) triflate catalysts

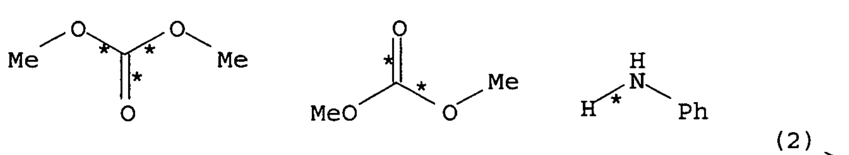
AUTHOR(S): Distaso, Monica; Quaranta, Eugenio
CORPORATE SOURCE: Dipartimento di Chimica, Universita di Bari, Bari, 70126, Italy

SOURCE: Journal of Catalysis (2004), 228(1), 36-42
CODEN: JCTLA5; ISSN: 0021-9517
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English

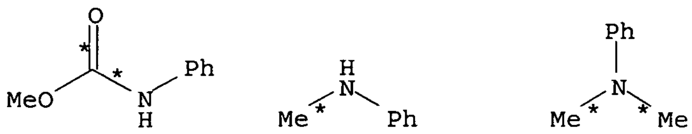
AB MeOCO2Ph (I) has been investigated as a carbomethoxylating agent for aromatic amines in the presence of Sc or La triflate catalysts. Under mild conditions (363 K), both Sc(OTf)3 and La(OTf)3 (OTf = O3SCF3) promote the carbamation of aniline and some industrially relevant aromatic diamines, such as 4,4'-methylenedianiline and 2,4-diaminotoluene, with I. Carbamate yield and selectivity are markedly affected by the exptl. conditions (temperature, reaction medium, nature of the metal center). Sc(OTf)3 is a more effective and selective carbamation catalyst than La(OTf)3. Ad hoc expts. have shown that, in the presence of the M(OTf)3 (M = Sc, La) catalysts, I is not only more reactive but also a more selective carbomethoxylating agent than (MeO)2CO.

REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(2) OF 12 4 **G** + 3 **B** ===> C + **D** + H



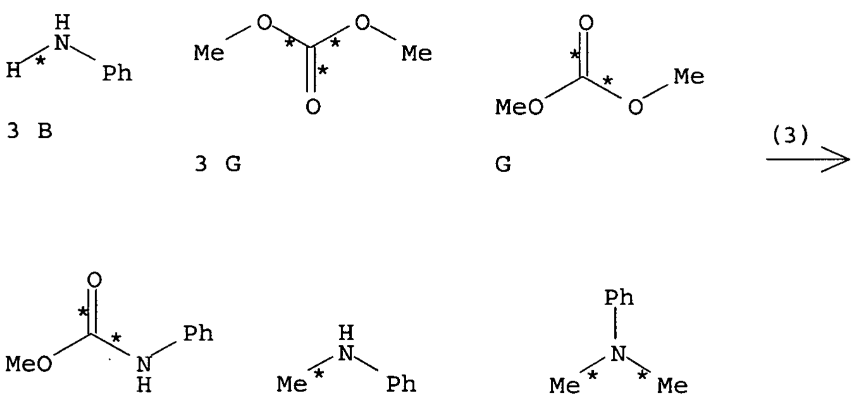
3 G G 3 B



C YIELD 3%   D YIELD 20%   H YIELD 5%

```
RX(2)       RCT  G 616-38-6, B 62-53-3
            PRO  C 2603-10-3, D 100-61-8, H 121-69-7
            CAT  52093-26-2 Methanesulfonic acid, trifluoro-, lanthanum(3+) salt
            CON  SUBSTAGE(1) 24 hours, 368K
                 SUBSTAGE(2) 368K -> room temperature
            NTE  no solvent
```

RX(3) OF 12   3 **B** + 4 **G** ===> C + **D** + H

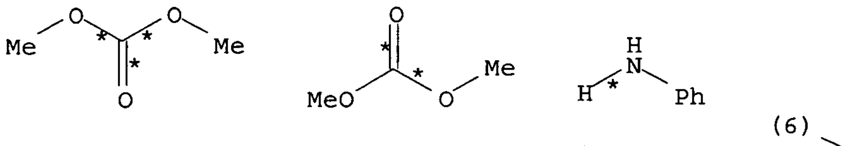


3 B   3 G   G

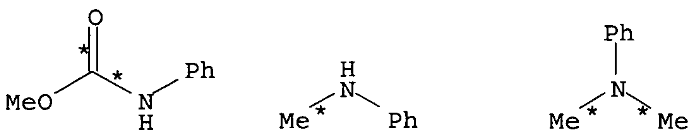
C YIELD 18%   D YIELD 20%   H YIELD 2%

```
RX(3)       RCT  B 62-53-3, G 616-38-6
            PRO  C 2603-10-3, D 100-61-8, H 121-69-7
            CAT  144026-79-9 Methanesulfonic acid, trifluoro-, scandium(3+) salt
            SOL  109-99-9 THF
            CON  27 hours, 363K
```

RX(6) OF 12   4 **G** + 3 **B** ===> C + **D** + H



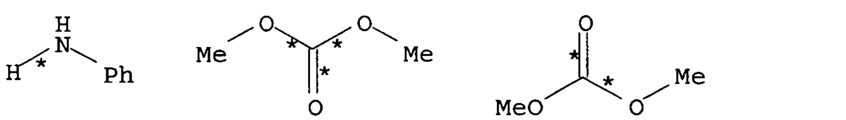
3 G G 3 B



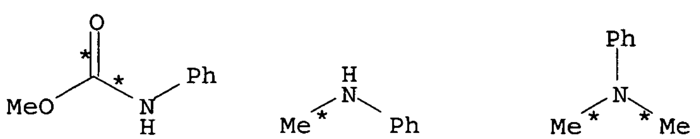
C	D	H
YIELD 8%	YIELD 25%	YIELD 10%

RX(6) RCT G **616-38-6**, B **62-53-3**
PRO C 2603-10-3, D **100-61-8**, H 121-69-7
CAT 144026-79-9 Methanesulfonic acid, trifluoro-, scandium(3+) salt
CON SUBSTAGE(1) 24 hours, 363K
SUBSTAGE(2) 363K -> room temperature
NTE no solvent

RX(7) OF 12 3 **B** + 4 **G** ===> C + **D** + H



3 B 3 G G



C	D	H
YIELD 7%	YIELD 26%	YIELD 5%

RX(7) RCT B **62-53-3**, G **616-38-6**
PRO C 2603-10-3, D **100-61-8**, H 121-69-7
CAT 52093-26-2 Methanesulfonic acid, trifluoro-, lanthanum(3+) salt
SOL 109-99-9 THF
CON 26 hours, 363K

L60 ANSWER 15 OF 44 CASREACT
ACCESSION NUMBER: 138:137040 CASREACT

TITLE: Process for the production of 1,2,4-triaminobenzene carbamic acid esters by amidation with pyrocarbonate or polypyrocarbonate esters
INVENTOR(S): Lankau, Hans-Joachim; Unverferth, Klaus; Arnold, Thomas; Schaefer, Jurgen; Meisel, Peter; Thiel, Wilfried
PATENT ASSIGNEE(S): Germany
SOURCE: U.S. Pat. Appl. Publ., 6 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2003023111	A1	20030130	US 2002-201296	20020724
DE 10136046	A1	20030213	DE 2001-10136046	20010725
WO 2003010134	A1	20030206	WO 2002-EP7301	20020702

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1414791	A1	20040506	EP 2002-790169	20020702

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK

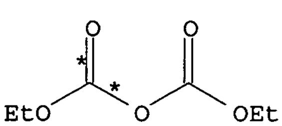
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2004536142	T2	20041202	JP 2003-515494	20020702

PRIORITY APPLN. INFO.: DE 2001-10136046 20010725
WO 2002-EP7301 20020702

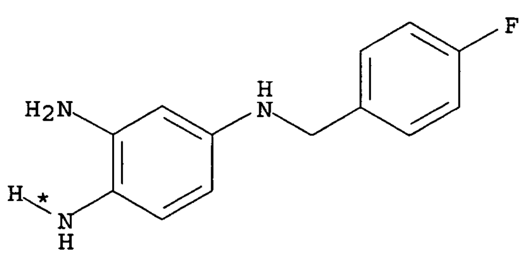
OTHER SOURCE(S): MARPAT 138:137040

AB A process for the production of 1,2,4-triaminobenzene carbamic acid esters is described which is characterized by the use of pyrocarbonate (e.g., Et polypyrocarbonate) or polypyrocarbonate esters. The use of pyrocarbonate or polypyrocarbonate esters brings about a rise in yield and increased purity of the product.

RX(1) OF 1 **A + B ===> C**

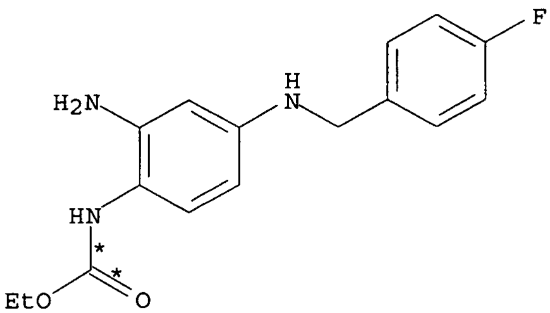


A



B

(1) →



C
YIELD 67%

RX(1) RCT A **1609-47-8**, B **491871-67-1**
PRO C **150812-12-7**
SOL 64-17-5 EtOH
CON SUBSTAGE(1) 2.5 hours, 6 - 11 deg C
SUBSTAGE(2) 3 hours, 6 - 11 deg C
SUBSTAGE(3) 11 deg C -> -5 deg C
SUBSTAGE(4) 2 hours, 5 - -5 deg C
NTE optimization study

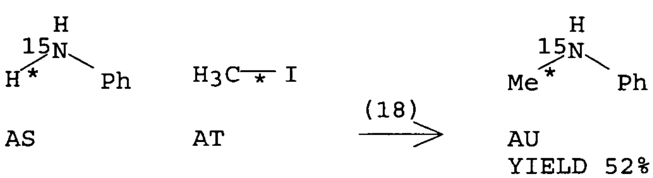
L60 ANSWER 16 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 140:164000 CASREACT
TITLE: Trans Influence on the Rate of Reductive Elimination. Reductive Elimination of Amines from Isomeric Arylpalladium Amides with Unsymmetrical Coordination Spheres
AUTHOR(S): Yamashita, Makoto; Vicario, Jose V. Cuevas; Hartwig, John F.
CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 06520-8107, USA
SOURCE: Journal of the American Chemical Society (2003), 125(52), 16347-16360
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB To determine the trans effect on the rates of reductive eliminations from arylpalladium(II) amido complexes, the reactions of arylpalladium amido complexes bearing sym. and unsym. DPPF (DPPF = bis(diphenylphosphino)ferrocene) ligands were studied. THF solns. of LPd(Ar)(NMeAr') (L = DPPF, DPPF-OMe, DPPF-CF3, DPPF-OMe,Ph, DPPF-Ph,CF3, and DPPF-OMe,CF3; Ar = C6H4-4-CF3; Ar' = C6H4-4-Me, Ph, and C6H4-4-OMe) underwent C-N bond forming reductive elimination at -15° to form the corresponding N-methyldiarylamine in high yield. Complexes ligated by sym. DPPF derivs. with electron-withdrawing substituents on the DPPF aryl groups underwent reductive elimination faster than complexes ligated by sym. DPPF derivs. with electron-donating substituents. Studies of arylpalladium amido complexes containing unsym. DPPF ligands revealed several trends. First, the complex with the weaker donor trans to nitrogen and the stronger donor trans to the palladium-bound aryl group underwent reductive elimination faster than the regioisomeric complex with the stronger donor trans to nitrogen and the weaker donor trans to the

palladium-bound aryl group. Second, the substituent effect of the phosphorus donor trans to the nitrogen was larger than the effect of the phosphorus donor trans to the palladium-bound aryl group. Third, the difference in rate between the isomeric arylpalladium amido complexes was similar in magnitude to the differences in rates resulting from conventional variation of substituents on the sym. phosphine ligands. This result suggests that the geometry of the complex is equal in importance to the donating ability of the dative ligands. The ratio of the differences in rates of reaction of the isomeric complexes was similar to the relative populations of the two geometric isomers. This result and consideration of transition state geometries suggest that the reaction rates are controlled more by substituent effects on ground state stability than on transition state energies. In addition, variation of the aryl group at the amido nitrogen showed systematically that complexes with more electron-donating groups at nitrogen undergo faster reductive elimination than those with less electron-donating groups at nitrogen.

REFERENCE COUNT: 111 THERE ARE 111 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(18) OF 88 **AS** + AT ===> **AU**...



RX(18) RCT AS **7022-92-6**

STAGE(1)
RGT AV **24424-99-5** (Boc)2O
SOL 109-99-9 THF, 7732-18-5 Water
CON 19 hours, room temperature

STAGE(2)
RGT AF 7732-18-5 Water
SOL 7732-18-5 Water

STAGE(3)
RCT AT 74-88-4
RGT AW 7646-69-7 NaH
SOL 68-12-2 DMF
CON 5 hours, room temperature

STAGE(4)
RGT AF 7732-18-5 Water
SOL 7732-18-5 Water

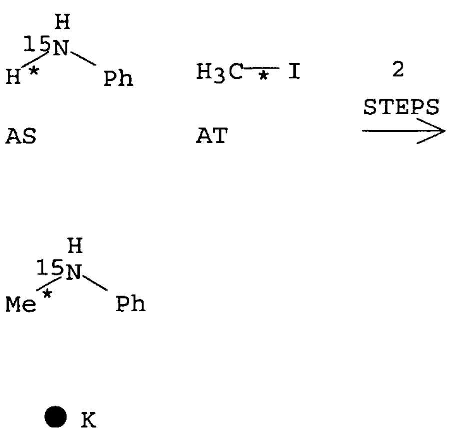
STAGE(5)
RGT AX 1493-13-6 F3CSO2H
SOL 75-09-2 CH2Cl2
CON 1.5 hours, room temperature

STAGE(6)
RGT AY 1310-73-2 NaOH

SOL 7732-18-5 Water

PRO AU **60317-46-6**

RX(44) OF 88 COMPOSED OF RX(18), RX(19)
RX(44) **AS** + AT ===> **BB**



BB
YIELD 44%

RX(18) RCT AS **7022-92-6**

STAGE(1)
RGT AV **24424-99-5** (Boc)2O
SOL 109-99-9 THF, 7732-18-5 Water
CON 19 hours, room temperature

STAGE(2)
RGT AF 7732-18-5 Water
SOL 7732-18-5 Water

STAGE(3)
RCT AT 74-88-4
RGT AW 7646-69-7 NaH
SOL 68-12-2 DMF
CON 5 hours, room temperature

STAGE(4)
RGT AF 7732-18-5 Water
SOL 7732-18-5 Water

STAGE(5)
RGT AX 1493-13-6 F3CSO2H
SOL 75-09-2 CH2Cl2
CON 1.5 hours, room temperature

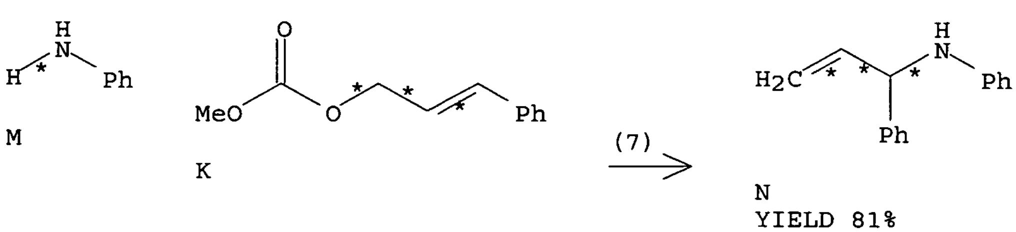
STAGE(6)
RGT AY 1310-73-2 NaOH
SOL 7732-18-5 Water

PRO AU 60317-46-6

RX(19) RCT AU 60317-46-6
RGT O 40949-94-8 K [N(SiMe3)2]
PRO BB **653588-24-0**
SOL 108-88-3 PhMe
CON 1 - 1.5 hour, room temperature

L60 ANSWER 17 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 140:59365 CASREACT
TITLE: Identification of an Activated Catalyst in the Iridium-Catalyzed Allylic Amination and Etherification. Increased Rates, Scope, and Selectivity
AUTHOR(S): Kiener, Christoph A.; Shu, Chutian; Incarvito, Christopher; Hartwig, John F.
CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 06520-8107, USA
SOURCE: Journal of the American Chemical Society (2003), 125(47), 14272-14273
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Studies were conducted to determine possible intermediates in the highly enantioselective, iridium-catalyzed amination and etherification of allylic carbonates, and these studies revealed that cyclometalation of the phosphoramidite ligand is likely to generate the active catalyst. The square-planar [Ir(COD)(L1)Cl] [L1 = (11bS)-N,N-bis[(1S)-1-phenylethyl]dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine] did not react with cinnamyl carbonate, but did react with amine to generate an Ir(I) trigonal bipyramidal complex coordinated by COD, a cyclometalated κ2-phosphoramidite, and a κ1-phosphoramidite. This complex reacted with phosphines to generate products from replacement of the κ1-phosphoramidite. These cyclometalated complexes were highly active catalysts for allylic amination and etherification and retained the high selectivity of the original catalyst system. In addition, these complexes combined with [Ir(COD)Cl]2 catalyzed reactions of amines with lower loadings, catalyzed reactions of alkylamines and aromatic amines that did not react with the original catalyst system, and catalyzed reactions of phenoxides under milder conditions.
REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

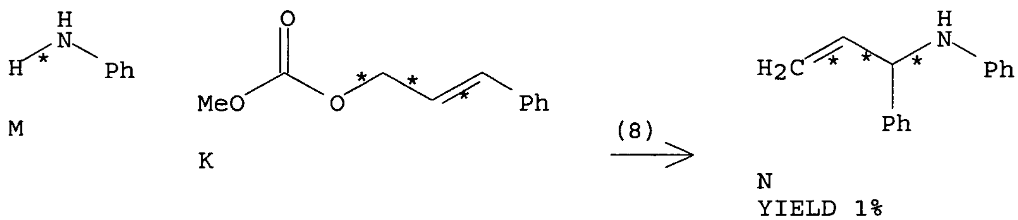
RX(7) OF 18 **M + K ===> N**



RX(7) RCT M **62-53-3**, K **85217-69-2**

PRO N **638166-19-5**
CAT 638166-14-0 Iridium, [(11bS)-N,
, 12112-67-3 Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-
cyclooctadiene]di-
SOL 109-99-9 THF
CON 2 hours, 25 deg C
NTE stereoselective

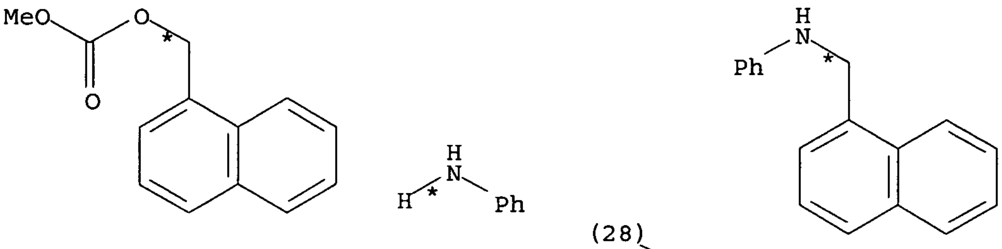
RX(8) OF 18 **M** + **K** ===> **N**



RX(8) RCT M **62-53-3**, K **85217-69-2**
PRO N **638166-19-5**
CAT 209482-27-9 Dinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-
amine, N,N-bis[(1S)-1-phenylethyl]-, (11bS)-, 12112-67-3
Iridium, di-μ-chlorobis[(1,2,5,6-η)-1,5-cyclooctadiene]di-
SOL 109-99-9 THF
CON 24 hours, 25 deg C
NTE stereoselective

L60 ANSWER 18 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 139:323314 CASREACT
TITLE: Palladium-Catalyzed Nucleophilic Benzylic Substitutions of Benzylic Esters
AUTHOR(S): Kuwano, Ryoichi; Kondo, Yutaka; Matsuyama, Yosuke
CORPORATE SOURCE: Department of Chemistry, Graduate School of Sciences, Kyushu University, Higashi-ku Fukuoka, 812-8581, Japan
SOURCE: Journal of the American Chemical Society (2003), 125(40), 12104-12105
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A palladium complex generated in situ from [Pd(η3-C3H5)(cod)]BF4 and DPPF is a good catalyst for benzylic alkylation of benzyl Me carbonate with the carbanion of di-Me malonates. The catalytic reaction is applicable to a wide range of the benzylations of benzylic esters with malonates. The catalytic activity was heavily affected by the bite angle of the bidentate phosphine ligand on palladium. DPEphos ligand is superior to DPPF in the case of palladium-catalyzed benzylic amination of benzylic esters.
REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(28) OF 28 **AK** + **AZ** ===> **BA**



AK AZ BA
YIELD 94%

RX(28) RCT AK **613670-57-8**, AZ **62-53-3**
PRO BA **7182-94-7**
CAT 166330-10-5 Phosphine, (oxydi-2,1-phenylene)bis[diphenyl-,
32915-11-0 Palladium(1+), [(1,2,5,6-η)-1,5-cyclooctadiene](η3-2-propenyl)-, tetrafluoroborate(1-)
SOL 110-71-4 (CH2OMe)2
CON 24 hours, 80 deg C

L60 ANSWER 19 OF 44 CASREACT

ACCESSION NUMBER: 139:70686 CASREACT

TITLE: Activity of amorphous V-AlPO4 and Co-AlPO4 in the selective synthesis of N-monoalkylated aniline via alkylation of aniline with methanol or dimethyl carbonate

AUTHOR(S): Nagaraju, N.; Kuriakose, George

CORPORATE SOURCE: Department of Chemistry, St. Joseph's College Post Graduate Center, Bangalore, 560 027, India

SOURCE: New Journal of Chemistry (2003), 27(4), 765-768
CODEN: NJCHE5; ISSN: 1144-0546

PUBLISHER: Royal Society of Chemistry

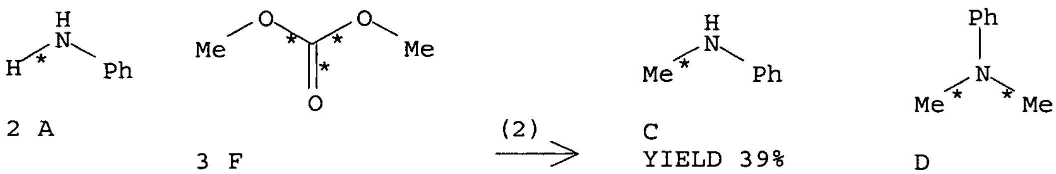
DOCUMENT TYPE: Journal

LANGUAGE: English

AB The vapor phase catalytic alkylation of aniline with methanol or di-Me carbonate (DMC) on amorphous AlPO4 and M-AlPO4 (M = Cu, Ni, Fe, Co and V) as catalysts was studied. Both V-AlPO4 and Co-AlPO4 showed high selectivity towards N-monomethylation of aniline. Further studies on the alkylation reactions were carried out using AlPO4 and M-AlPO4 (M = V and Co) as catalysts at different temps. and molar ratio of aniline to methanol or DMC. V-AlPO4 showed 100% selectivity for N-monomethylation of aniline, irresp. of the temperature or the molar ratio of the reactants. Co-AlPO4 showed 100% selectivity for N-monomethylated product only when the aniline-to-methanol (or DMC) molar ratio is 2:1. The aniline conversion towards N-methylaniline(NMA) was 39%. DMC was a better methylating agent than methanol with respect to the percentage conversion of aniline to N-methylaniline.

REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(2) OF 2 2 **A** + 3 **F** ===> **C** + D



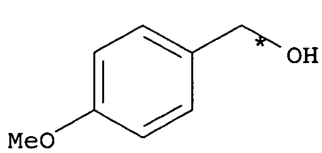
RX(2) RCT A **62-53-3**, F **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 501936-33-0 Phosphoric acid, aluminum vanadium salt
CON 523K
NTE gas phase, down-flow fixed-bed glass reactor used, yield depends on reaction conditions, thermal

L60 ANSWER 20 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 141:190769 CASREACT
TITLE: Intrinsic sensing fluorescent probe for the solid phase synthesis of 1,4-benzodiazepine-2,5-dione
AUTHOR(S): Rivero, Ignacio A.; Peralta, Margarita; Heredia, Socorro; Madrigal, Domingo; Pina-Luis, Georgina; Chavez, Daniel
CORPORATE SOURCE: Centro de Graduados e Investigacion, Instituto Tecnologico de Tijuana, Tijuana, 22000, Mex.
SOURCE: ARKIVOC (Gainesville, FL, United States) (2003), (11), 27-36
CODEN: AGFUAR
URL: http://www.arkat-usa.org/ark/journal/2003/Regional_Issue/MX-827E/827E.pdf
PUBLISHER: Arkat USA Inc.
DOCUMENT TYPE: Journal; (online computer file)
LANGUAGE: English

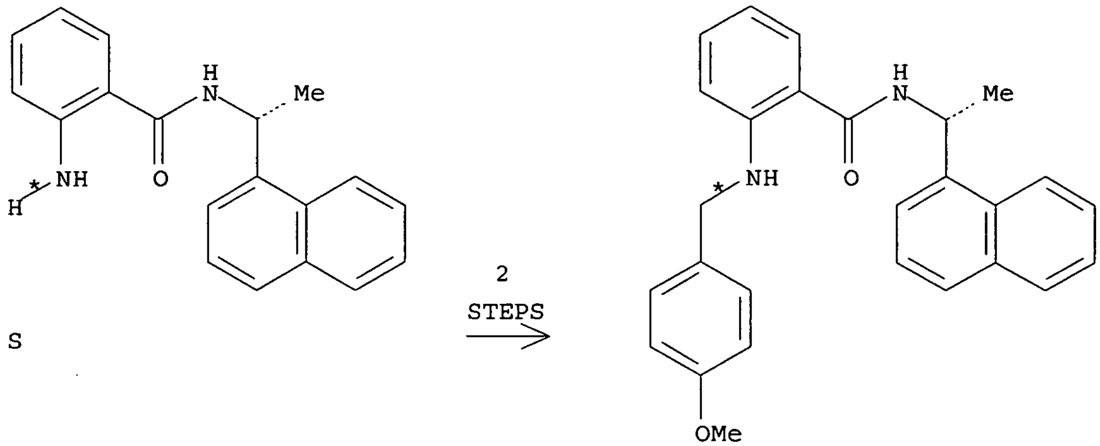
AB The synthesis of 4-N-naphthylethyl-1,4-benzodiazepine-2,5-dione supported on solid phase is described. The fluorescent naphthyl group is used as internal sensor for monitoring various stages on the synthetic reaction by spectral differences in fluorescence. Our results show the efficacy of fluorescence as a direct method for the evaluation of reaction progress. This method is fast, sensitive and non-destructive. A target mol. was synthesized which could be used in the synthesis of a variety of benzodiazepines.

REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(60) OF 162 COMPOSED OF RX(7), RX(8)
RX(60) Q + **S** ===> **T**



Q



S

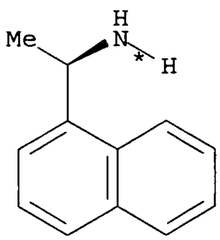
T
YIELD 83%

```
RX(7)       RCT  Q 105-13-5D

              STAGE(1)
                 SOL   75-09-2 CH2Cl2
                 CON   30 minutes, room temperature

              STAGE(2)
                 RGT   L 603-35-0 PPh3
                 SOL   75-09-2 CH2Cl2
                 CON   5 minutes, 0 deg C

              STAGE(3)
                 RGT   M 32315-10-9 (Cl3CO)2CO
                 CON   5 minutes, 0 deg C

              STAGE(4)
                 SOL   75-09-2 CH2Cl2
                 CON   room temperature

              STAGE(5)
                 SOL   75-09-2 CH2Cl2
                 CON   1 hour, room temperature

            PRO  R 824-94-2D
            NTE  solid-supported reaction, crude from stage 2,3 added in stage 4

RX(8)       RCT  R 824-94-2D

              STAGE(1)
                 SOL   68-12-2 DMF
                 CON   30 minutes, room temperature

              STAGE(2)
                 RCT   S 737765-26-3
                 RGT   U 584-08-7 K2CO3
                 CON   SUBSTAGE(1) room temperature
                       SUBSTAGE(2) 12 hours, 80 deg C
```

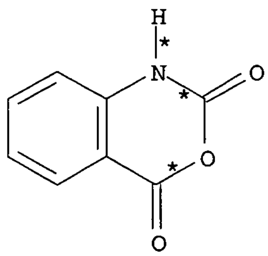
PRO T **737765-27-4D**
NTE solid-supported reaction

RX(97) OF 162 COMPOSED OF REACTION SEQUENCE RX(16), RX(8)
AND REACTION SEQUENCE RX(7), RX(8)

...Z + AA ===> S...
...Q + **S** ===> **T**

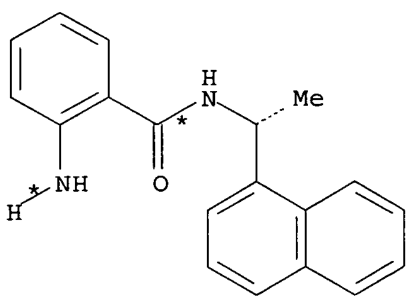


Z



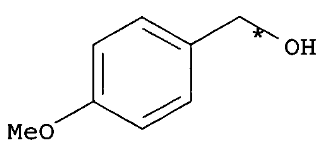
AA

2
STEPS

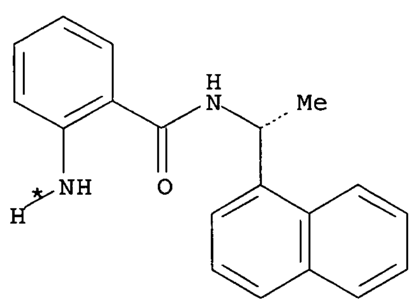


S

START NEXT REACTION SEQUENCE

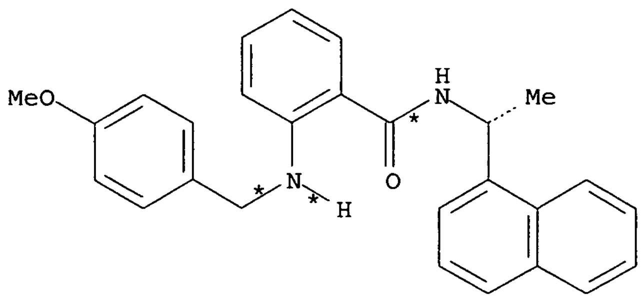


Q



S

2
STEPS



T
YIELD 83%

```
RX(16)     RCT  Z 3886-70-2, AA 118-48-9

             STAGE(1)
                SOL  68-12-2 DMF
                CON  SUBSTAGE(1) 20 minutes, 55 deg C
                     SUBSTAGE(2) 2 hours, 55 - 60 deg C

             STAGE(2)
                SOL  7732-18-5 Water

           PRO  S 737765-26-3

RX(7)      RCT  Q 105-13-5D

             STAGE(1)
                SOL  75-09-2 CH2Cl2
                CON  30 minutes, room temperature

             STAGE(2)
                RGT  L 603-35-0 PPh3
                SOL  75-09-2 CH2Cl2
                CON  5 minutes, 0 deg C

             STAGE(3)
                RGT  M 32315-10-9 (Cl3CO)2CO
                CON  5 minutes, 0 deg C

             STAGE(4)
                SOL  75-09-2 CH2Cl2
                CON  room temperature

             STAGE(5)
                SOL  75-09-2 CH2Cl2
                CON  1 hour, room temperature

           PRO  R 824-94-2D
           NTE  solid-supported reaction, crude from stage 2,3 added in stage 4

RX(8)      RCT  R 824-94-2D

             STAGE(1)
                SOL  68-12-2 DMF
```

CON 30 minutes, room temperature

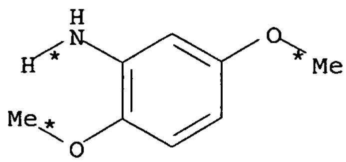
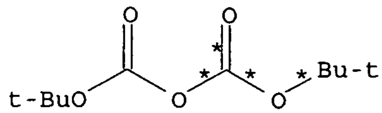
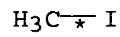
STAGE(2)
RCT S **737765-26-3**
RGT U 584-08-7 K2CO3
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) 12 hours, 80 deg C

PRO T **737765-27-4D**
NTE solid-supported reaction

L60 ANSWER 21 OF 44 CASREACT

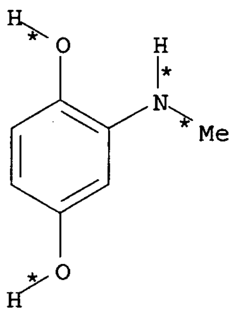
ACCESSION NUMBER: 139:373878 CASREACT
TITLE: Molecular design and biological activities of protein-tyrosine phosphatase inhibitors
AUTHOR(S): Umezawa, Kazuo; Kawakami, Mariko; Watanabe, Takumi
CORPORATE SOURCE: Faculty of Science and Technology, Department of Applied Chemistry, Keio University, Kohoku-ku, Yokohama, 223-0061, Japan
SOURCE: Pharmacology & Therapeutics (2003), 99(1), 15-24
CODEN: PHTHDT; ISSN: 0163-7258
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal; General Review
LANGUAGE: English
AB A review. Protein-tyrosine kinase (PTKase) and protein-tyrosine phosphatase (PTPase) regulate the intracellular signal transduction in various biol. processes. PTPase often neg. regulates the intracellular protein-tyrosine phosphorylation. PTPases are considered to be involved in the etiol. of diabetes mellitus and neural diseases, such as Alzheimer's disease and Parkinson's disease. Therefore, PTPase inhibitors should be useful tools to study the role of PTPases in these diseases and other biol. phenomena, and they hopefully may be developed into chemotherapeutic agents. We first discovered a naturally occurring PTPase inhibitor, dephostatin, in 1993. Later, we developed stable and safe dephostatin analogs by a mol. design approach employing the concept of CH/π interaction. We prepared Et-3,4-dephostatin as a stable analog and found it to inhibit PTP-1B and SHPTP-1 PTPases selectively. Et-3,4-dephostatin increased the tyrosine phosphorylation of the insulin receptor and insulin receptor substrate-1 (IRS-1), with or without insulin, in differentiated 3T3-L1 mouse adipocytes. It also increased the phosphorylation and activation of Akt. The analog also enhanced translocation of glucose transporter 4 (GLUT4) from the cytoplasm to the membrane and 2-deoxyglucose transport. It also showed an in vivo antidiabetic effect in terms of reducing the high blood glucose level in KK-Ay mice after oral administration. Since Et-3,4-dephostatin contains a nitrosamine moiety, we designed nitrosamine-free dephostatin analogs employing the concept of CH/π interaction. Then, we synthesized methoxime- and hexyl-methoxime-3,4-dephostatin as nitrosamine-free analogs. These analogs also showed antidiabetic activity in vivo and illustrate the utility of the CH/π interaction mol. design approach.
REFERENCE COUNT: 41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(19) OF 26 COMPOSED OF RX(1), RX(2), RX(3)
RX(19) **A** + **B** + G ===> **K**

A B G

3 STEPS
--->



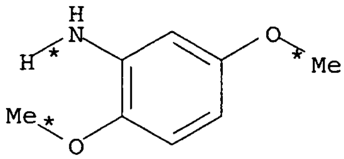
K

RX(1) RCT A **102-56-7**, B **24424-99-5**
RGT D 121-44-8 Et3N
PRO C 170959-02-1
SOL 7732-18-5 Water, 123-91-1 Dioxane

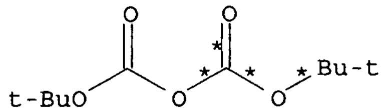
RX(2) RCT C 170959-02-1, G 74-88-4
RGT I 7646-69-7 NaH
PRO H 155853-12-6
SOL 68-12-2 DMF

RX(3) RCT H 155853-12-6
RGT L 10294-33-4 BBr3
PRO K **155853-13-7**
SOL 75-09-2 CH2Cl2

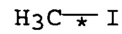
RX(20) OF 26 COMPOSED OF RX(1), RX(2), RX(11)
RX(20) **A** + **B** + G + 2 AJ ===> **N**



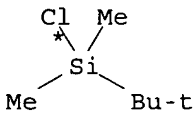
A



B

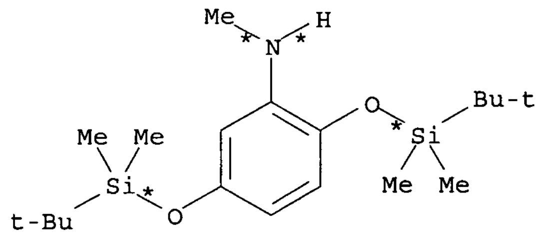


G



2 AJ

3
STEPS



N
YIELD 41%

```
RX(1)      RCT  A 102-56-7, B 24424-99-5
           RGT  D 121-44-8 Et3N
           PRO  C 170959-02-1
           SOL  7732-18-5 Water, 123-91-1 Dioxane

RX(2)      RCT  C 170959-02-1, G 74-88-4
           RGT  I 7646-69-7 NaH
           PRO  H 155853-12-6
           SOL  68-12-2 DMF

RX(11)     RCT  H 155853-12-6

             STAGE(1)
                RGT  L 10294-33-4 BBr3
                SOL  75-09-2 CH2Cl2

             STAGE(2)
                RCT  AJ 18162-48-6
                RGT  AK 288-32-4 1H-Imidazole
                SOL  68-12-2 DMF

           PRO  N 155853-14-8
```

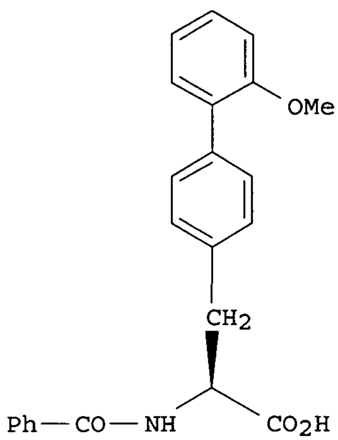
L60 ANSWER 22 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

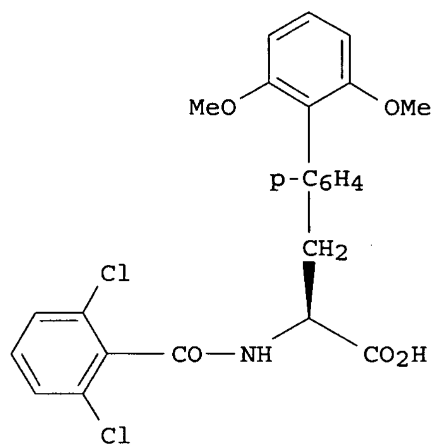
ACCESSION NUMBER: 137:263272 CASREACT

TITLE: Synthesis and SAR of N-Benzoyl-L-Biphenylalanine Derivatives: Discovery of TR-14035, A Dual α4β7/α4β1 Integrin Antagonist

AUTHOR(S): Sircar, Ila; Gudmundsson, Kristjan S.; Martin, Richard; Liang, Jimmy; Nomura, Sumihiro; Jayakumar, Honnappa; Teegarden, Bradley R.; Nowlin, Dawn M.; Cardarelli, Pina M.; Mah, Jason R.; Connell, Samuel; Griffith, Ronald C.; Lazarides, Elias

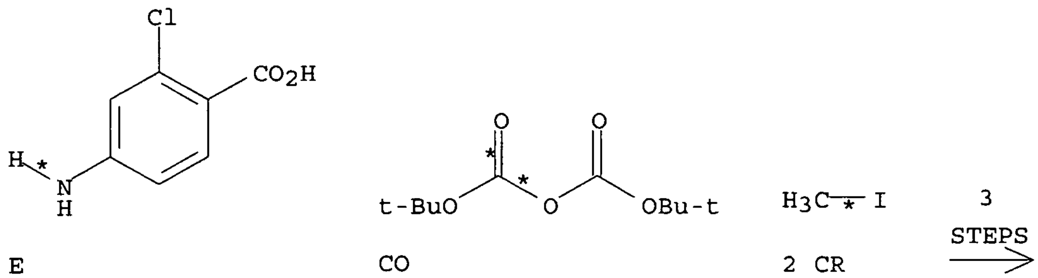
CORPORATE SOURCE: Department of Chemical Science, Tanabe Research Laboratories USA, Inc., San Diego, CA, 92121, USA
SOURCE: Bioorganic & Medicinal Chemistry (2002), 10(6), 2051-2066
CODEN: BMECEP; ISSN: 0968-0896
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
GI


I

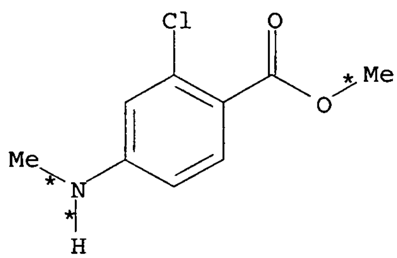

II

AB α4β1 And α4β7 integrins are key regulators of physiol. and pathol. responses in inflammation and autoimmune disease. The effectiveness of anti-integrin antibodies to attenuate a number of inflammatory/immune conditions provides a strong rationale to target integrins for drug development. Important advances have been made in identifying potent and selective candidates, peptides and peptidomimetics, for further development. Herein, we report the discovery of a series of novel N-benzoyl-L-biphenylalanine derivs. that are potent inhibitors of α4 integrins. The potency of the initial lead compound (I: IC50 α4β7/α4β1=5/33 μM) was optimized via sequential manipulation of substituents to generate low nM, orally bioavailable dual α4β1/α4β7 antagonists. The SAR also led to the identification of several subnanomolar antagonists. Compound (II) (TR-14035; IC50 α4β7/α4β1=7/87 nM) has completed Phase I studies in Europe. The synthesis, SAR and biol. evaluation of these compds. are described.
REFERENCE COUNT: 39 THERE ARE 39 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(224) OF 267 COMPOSED OF RX(42), RX(43), RX(4)
RX(224) **E** + **CO** + 2 CR ===> **Q**

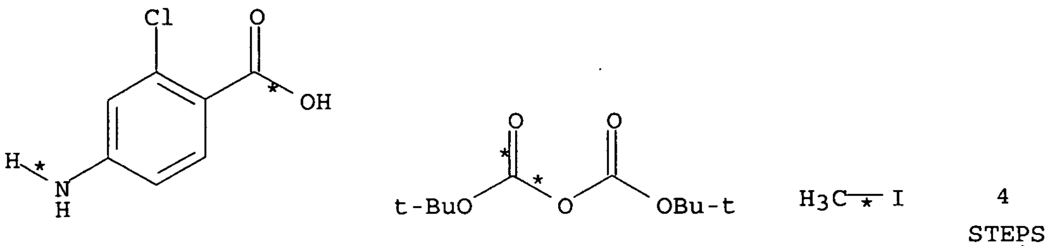


E CO 2 CR

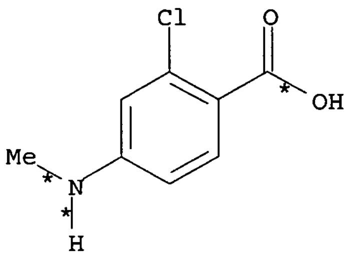


Q

```
RX(42)     RCT  E 2457-76-3, CO 24424-99-5
           RGT  DJ 1310-73-2 NaOH
           PRO  DO 232275-73-9
           SOL  123-91-1 Dioxane

RX(43)     RCT  DO 232275-73-9

             STAGE(1)
                RGT  DP 124-41-4 NaOMe
                SOL  68-12-2 DMF

             STAGE(2)
                RCT  CR 74-88-4

           PRO  P 232275-74-0

RX(4)      RCT  P 232275-74-0
           RGT  B 76-05-1 F3CCO2H
           PRO  Q 204972-82-7
           SOL  75-09-2 CH2Cl2


RX(227) OF 267 COMPOSED OF RX(42), RX(43), RX(4), RX(44)
RX(227)   E  +  CO  +  2 CR  ===>  DQ
```



E CO 2 CR



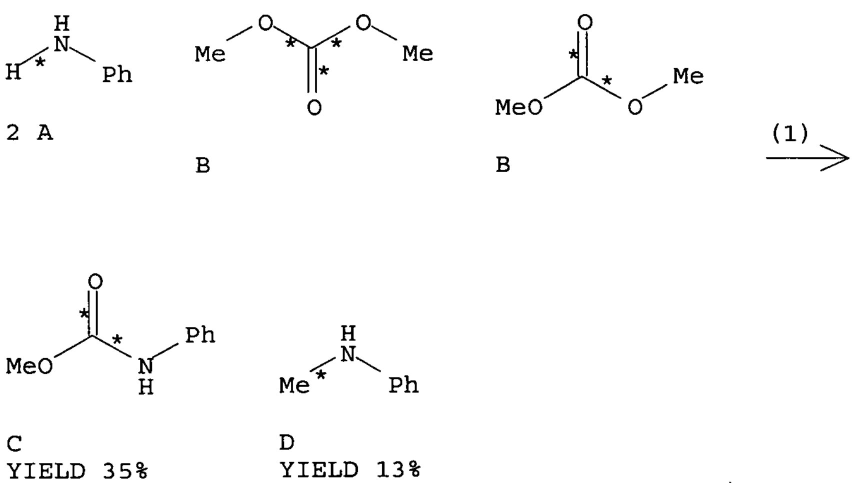
DQ

```
RX(42)     RCT  E 2457-76-3, CO 24424-99-5
           RGT  DJ 1310-73-2 NaOH
           PRO  DO 232275-73-9
           SOL  123-91-1 Dioxane

RX(43)     RCT  DO 232275-73-9

             STAGE(1)
                RGT  DP 124-41-4 NaOMe
                SOL  68-12-2 DMF

             STAGE(2)
                RCT  CR 74-88-4

           PRO  P 232275-74-0

RX(4)      RCT  P 232275-74-0
           RGT  B 76-05-1 F3CCO2H
           PRO  Q 204972-82-7
           SOL  75-09-2 CH2Cl2

RX(44)     RCT  Q 204972-82-7
           RGT  AV 1310-65-2 LiOH
           PRO  DQ 3975-62-0
           SOL  109-99-9 THF, 67-56-1 MeOH
```

L60 ANSWER 23 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 138:237878 CASREACT
TITLE: A new catalyst for the synthesis of N,N-biphenylurea from aniline and dimethyl carbonate

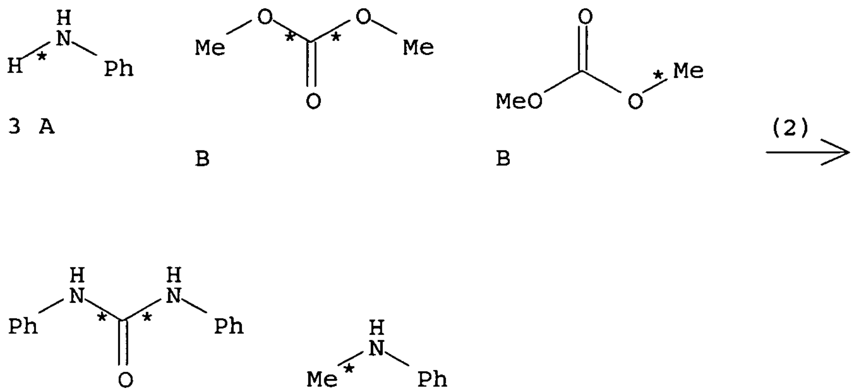
AUTHOR(S): Nagaraju, N.; Kuriakose, George
CORPORATE SOURCE: Department of Chemistry, St.Joseph's College Post Graduate Center, Shanthi Nagar, Bangalore, 560 027, India
SOURCE: Green Chemistry (2002), 4(3), 269-271
CODEN: GRCHFJ; ISSN: 1463-9262
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
AB An environmentally benign method for the synthesis of biphenylurea (BPU) in the presence of ecofriendly catalysts was reported. Catalytic activity of various metal aluminophosphates, M-AlPO4 (M = V, Fe, Co, Ni and Cu) and cobalt supported on hydrated Al2O3, SiO2 and ZrO2 was studied in the synthesis of BPU from aniline and di-Me carbonate (DMC). All the catalysts used are amorphous. The catalytic activity studies were carried out in the liquid phase under refluxing conditions. Formation of BPU is found to depend on the type of the catalyst, the molar ratio of aniline to DMC and the duration of the reaction. N-Methylaniline and Me N-phenylcarbamate are formed as byproducts of the reaction. Of all the catalysts used, Co-AlPO4 was found to yield the highest percentage of BPU whereas cobalt supported hydrated Al2O3, SiO2 and ZrO2 failed to give any BPU.
REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 2 2 **A** + 2 **B** ===> C + **D**



RX(1) RCT A **62-53-3**, B **616-38-6**
PRO C 2603-10-3, D **100-61-8**
CAT 23209-38-3 Phosphoric acid, aluminum cobalt salt
CON 4 hours, reflux
NTE green chem.-waste redn., optimization study

RX(2) OF 2 3 **A** + 2 **B** ===> F + **D**



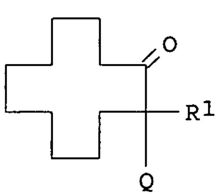
3 A B B

F
YIELD 68%

D
YIELD 24%

RX(2) RCT A **62-53-3**, B **616-38-6**
PRO F 102-07-8, D **100-61-8**
CAT 23209-38-3 Phosphoric acid, aluminum cobalt salt
CON 4 hours, reflux
NTE green chem.-waste redn., optimization study

L60 ANSWER 24 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 137:46974 CASREACT
TITLE: Synthesis and biological activity of 2-arylaminomethyl-2-ethoxycarbonylfcyclododecanone
AUTHOR(S): Qi, Chuan-min; Zhang, Guan-xin; Wang, Yun-feng
CORPORATE SOURCE: Department of Chemistry, Beijing Normal University, Beijing, 100875, Peop. Rep. China
SOURCE: Yingyong Huaxue (2002), 19(3), 243-246
CODEN: YIHUED; ISSN: 1000-0518
PUBLISHER: Yingyong Huaxue Bianji Weiyuanhui
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
GI

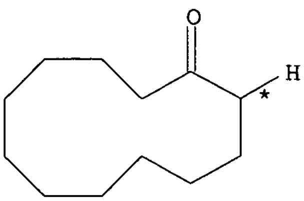


II

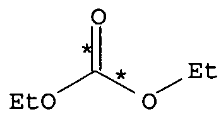
AB Eight title Mannich bases I (R = H, 4-Cl, 2-Cl, 4-CH3O, 2-Br, 4-Br, 4-CH3; R1 = COOCH2CH3; Q = CH2Q1; Q1 = NHC6H4R) were synthesized from reaction of formaldehyde and dimethylamine hydrochloride and Q1H with I (Q = CH2N(CH3)2·HCl; R1 = COOCH2CH3), which was prepared from I (R1 = H; Q = H). Their structures were confirmed by IR, 1H NMR and elemental anal. Mannich reaction of an amine was occurred at ortho position of 2-ethoxycarbonyl-cyclododecanone. The preliminary in vitro bioassays of

some compds. indicated that some of compds. IV showed certain fungistatic activity against plant pathogens. 4-Br substituted Ph aminomethyl-2-ethoxycarbonyl-cyclododecanone exerted appreciable inhibitory effect on xylene-induced ear edema in mice.

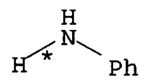
RX(11) OF 17 COMPOSED OF RX(1), RX(3)
RX(11)   A + **B** + **L** + G ===> **M**



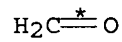
A



B

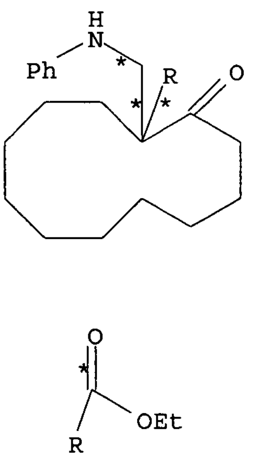


L



G

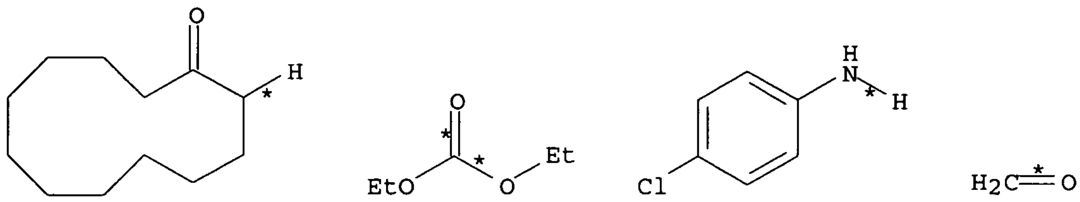
2
STEPS
⟶



M
YIELD 57%

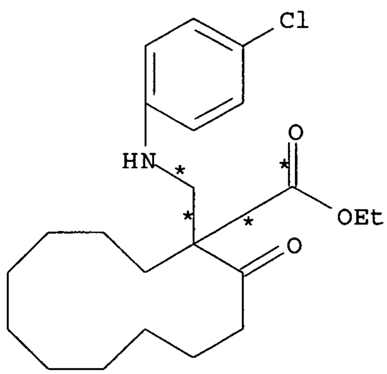
RX(1)	RCT	A 830-13-7, B **105-58-8**
	RGT	D 7646-69-7 NaH
	PRO	C 4017-60-1
	SOL	71-43-2 Benzene
	NTE	reflux, 6 h
RX(3)	RCT	C 4017-60-1, L **62-53-3**, G 50-00-0
	PRO	M **438051-08-2**
	SOL	64-17-5 EtOH
	NTE	50°, 9 h

RX(12) OF 17 COMPOSED OF RX(1), RX(4)
RX(12) A + **B** + **N** + G ===> **O**



A B N G

2
STEPS
--->

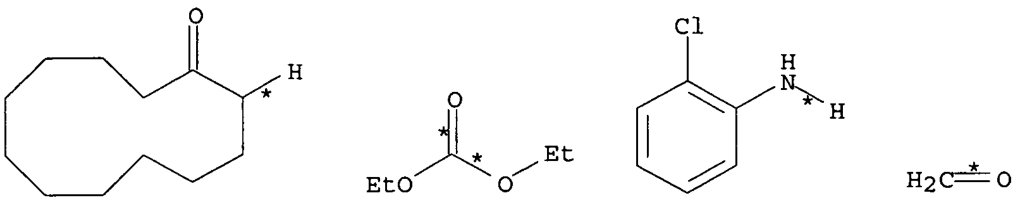


O
YIELD 49%

RX(1) RCT A 830-13-7, B **105-58-8**
RGT D 7646-69-7 NaH
PRO C 4017-60-1
SOL 71-43-2 Benzene
NTE reflux, 6 h

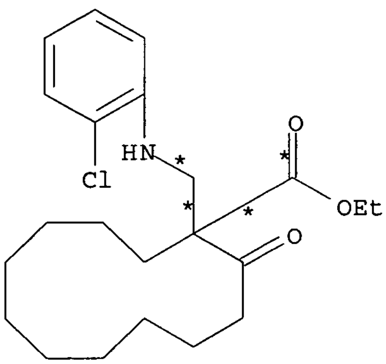
RX(4) RCT N **106-47-8**, C 4017-60-1, G 50-00-0
PRO O **438051-10-6**
SOL 64-17-5 EtOH
NTE room temp.

RX(13) OF 17 COMPOSED OF RX(1), RX(5)
RX(13) A + **B** + **P** + G ===> **Q**



A B P G

2
STEPS

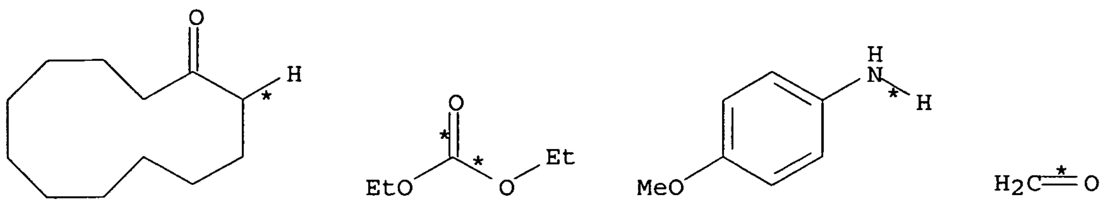


Q
YIELD 42%

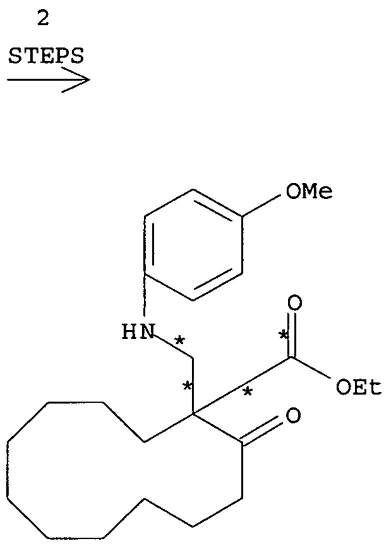
RX(1) RCT A 830-13-7, B **105-58-8**
RGT D 7646-69-7 NaH
PRO C 4017-60-1
SOL 71-43-2 Benzene
NTE reflux, 6 h

RX(5) RCT P **95-51-2**, C 4017-60-1, G 50-00-0
PRO Q **438051-12-8**
SOL 64-17-5 EtOH
NTE room temp.

RX(14) OF 17 COMPOSED OF RX(1), RX(6)
RX(14) A + **B** + **R** + G ===> **S**



A B R G

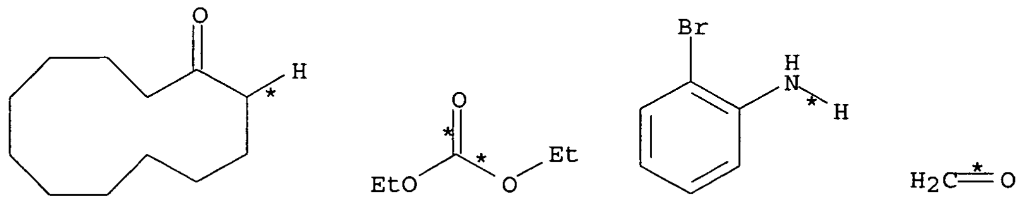


S
YIELD 65%

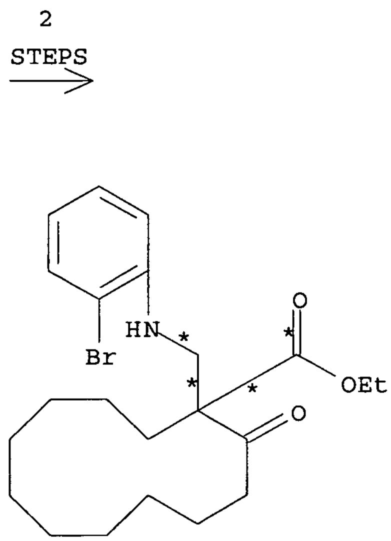
RX(1) RCT A 830-13-7, B **105-58-8**
RGT D 7646-69-7 NaH
PRO C 4017-60-1
SOL 71-43-2 Benzene
NTE reflux, 6 h

RX(6) RCT R **104-94-9**, C 4017-60-1, G 50-00-0
PRO S **438051-13-9**
SOL 64-17-5 EtOH
NTE room temp.

RX(15) OF 17 COMPOSED OF RX(1), RX(7)
RX(15) A + **B** + **T** + G ===> **U**



A B T G

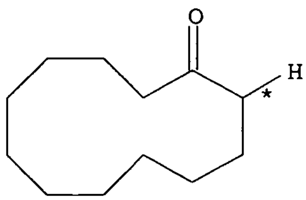


U
YIELD 43%

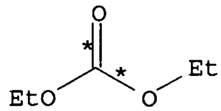
RX(1) RCT A 830-13-7, B **105-58-8**
RGT D 7646-69-7 NaH
PRO C 4017-60-1
SOL 71-43-2 Benzene
NTE reflux, 6 h

RX(7) RCT T **615-36-1**, C 4017-60-1, G 50-00-0
PRO U **438051-14-0**
SOL 64-17-5 EtOH
NTE room temp.

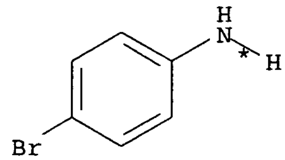
RX(16) OF 17 COMPOSED OF RX(1), RX(8)
RX(16) A + **B** + **V** + G ===> **W**



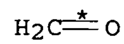
A



B

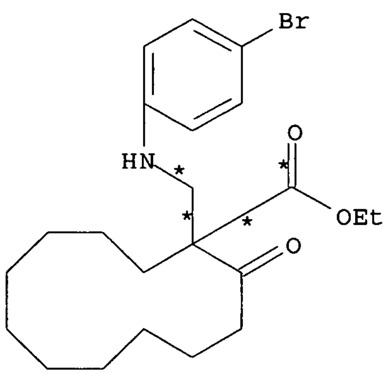


V



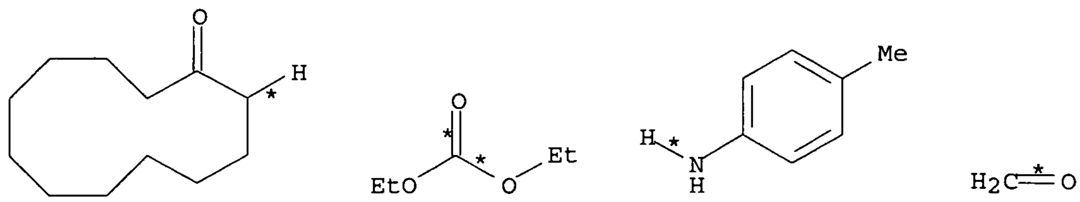
G

2
STEPS



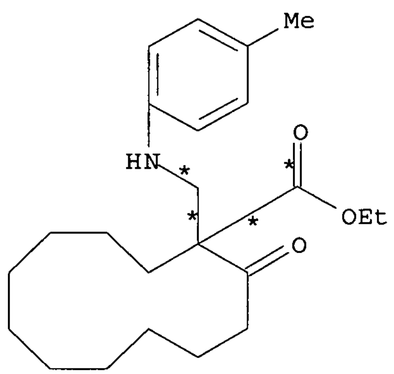
W
YIELD 50%

```
RX(1)      RCT  A 830-13-7, B 105-58-8
           RGT  D 7646-69-7 NaH
           PRO  C 4017-60-1
           SOL  71-43-2 Benzene
           NTE  reflux, 6 h

RX(8)      RCT  V 106-40-1, C 4017-60-1, G 50-00-0
           PRO  W 438051-15-1
           SOL  64-17-5 EtOH
           NTE  room temp.


RX(17) OF 17 COMPOSED OF RX(1), RX(9)
RX(17)     A  +  B  +  X  +  G  ===>  Y
```



A B X G

2
STEPS



Y
YIELD 62%

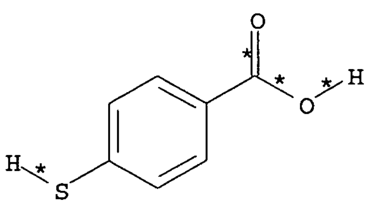
RX(1) RCT A 830-13-7, B **105-58-8**
RGT D 7646-69-7 NaH
PRO C 4017-60-1
SOL 71-43-2 Benzene
NTE reflux, 6 h

RX(9) RCT X **106-49-0**, C 4017-60-1, G 50-00-0
PRO Y **438051-17-3**
SOL 64-17-5 EtOH
NTE room temp.

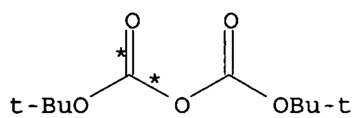
L60 ANSWER 25 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 136:167326 CASREACT
TITLE: Design and Synthesis of Potent Nonpeptidic Farnesyltransferase Inhibitors Based on a Terphenyl Scaffold
AUTHOR(S): Ohkanda, Junko; Lockman, Jeffrey W.; Kothare, Mohit A.; Qian, Yimin; Blaskovich, Michelle A.; Sebti, Said M.; Hamilton, Andrew D.
CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 06520-8107, USA

SOURCE: Journal of Medicinal Chemistry (2002), 45(1), 177-188
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
AB By modification of key carboxylate, hydrophobic, and zinc-binding groups projected from a sterically restricted terphenyl scaffold, a series of simple and nonpeptide mimetics of the Cys-Val-Ile-Met tetrapeptide substrate of protein farnesyltransferase (FTase) have been designed and synthesized. A crystal structure of 4-nitro-2-phenyl-3'-methoxycarbonylbiphenyl shows that the terphenyl fragment provides a large hydrophobic surface that potentially mimics the hydrophobic side chains of the three terminal residues in the tetrapeptide. 2-Phenyl-3-{N-[1-(4-cyanobenzyl)-1H-imidazol-5-yl]methyl}amino-3'-carboxylbiphenyl, in which the free thiol group was replaced with a 1-(4-cyanobenzyl)imidazole group, shows submicromolar inhibition activity against FTase in vitro and inhibits H-Ras processing in whole cells.
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

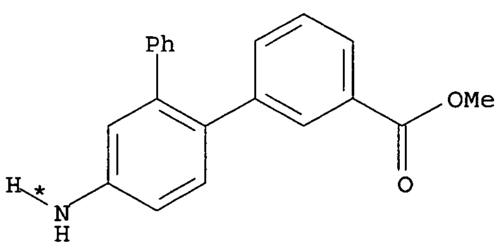
RX(189) OF 350 COMPOSED OF RX(30), RX(31), RX(32), RX(33)
RX(189) CE + **CF** + **AQ** ===> **BK**



CE

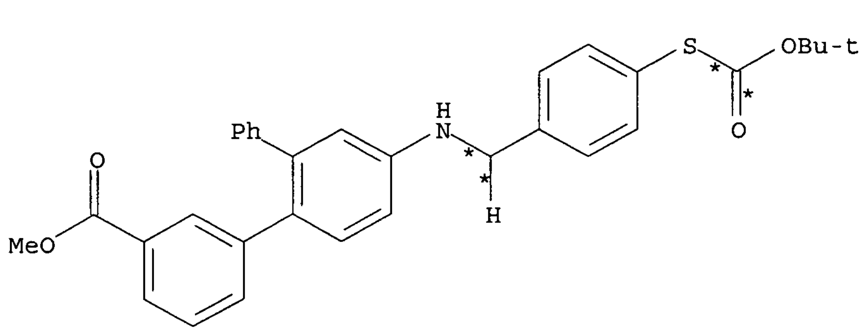


CF



AQ

4
STEPS
⟶

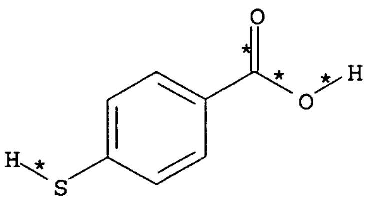


BK
YIELD 78%

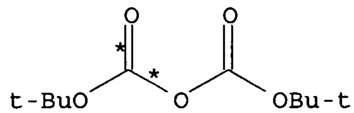
```
RX(30)     RCT  CE 1074-36-8, CF 24424-99-5
           RGT  CA 121-44-8 Et3N
           PRO  CG 396725-66-9
           CAT  1122-58-3 4-DMAP
           SOL  109-99-9 THF

RX(31)     RCT  CG 396725-66-9

             STAGE(1)
                RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
                SOL  109-99-9 THF

             STAGE(2)
                RGT  CK 16940-66-2 NaBH4

             STAGE(3)
                RGT  O 7647-01-0 HCl
                SOL  7732-18-5 Water

           PRO  CI 396725-67-0

RX(32)     RCT  CI 396725-67-0

             STAGE(1)
                RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
                SOL  75-09-2 CH2Cl2

             STAGE(2)
                RGT  CA 121-44-8 Et3N

           PRO  CL 396725-68-1

RX(33)     RCT  CL 396725-68-1, AQ 396725-50-1

             STAGE(1)
                CAT  64-19-7 AcOH
                SOL  75-09-2 CH2Cl2

             STAGE(2)
                RGT  CK 16940-66-2 NaBH4

           PRO  BK 396725-69-2
```

NTE in-situ generated reactant

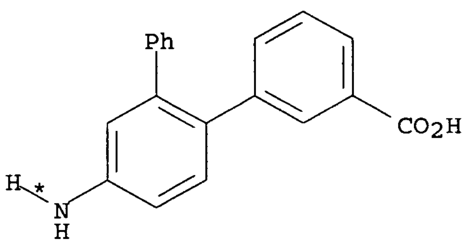
RX(190) OF 350 COMPOSED OF RX(30), RX(31), RX(32), RX(34)
RX(190) CE + **CF** + **AT** ===> **BH**



CE

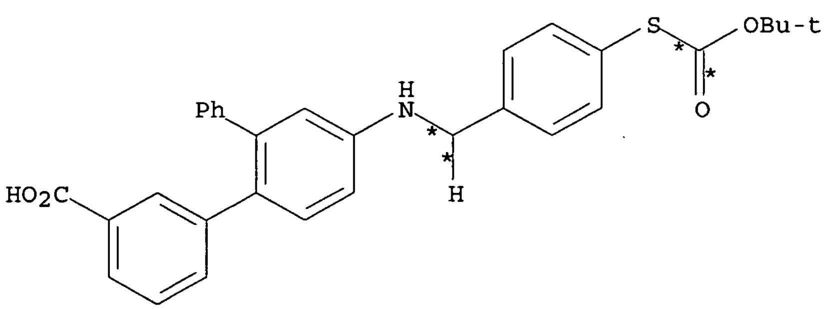


CF



AT

4
STEPS
→



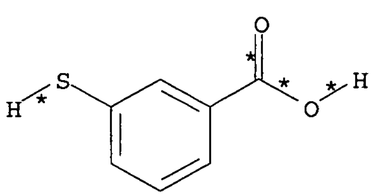
BH
YIELD 23%

RX(30) RCT CE 1074-36-8, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CG 396725-66-9
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

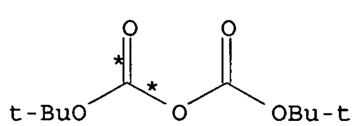
RX(31) RCT CG 396725-66-9

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et

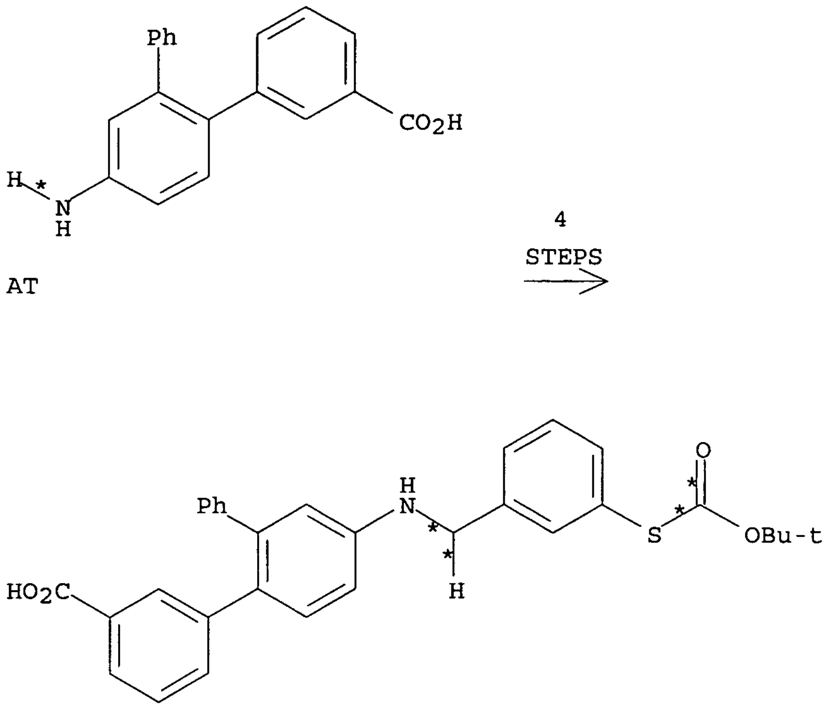
```
          SOL  109-99-9 THF

        STAGE(2)
          RGT  CK 16940-66-2 NaBH4

        STAGE(3)
          RGT  O 7647-01-0 HCl
          SOL  7732-18-5 Water

     PRO  CI 396725-67-0

RX(32)    RCT  CI 396725-67-0

        STAGE(1)
          RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
          SOL  75-09-2 CH2Cl2

        STAGE(2)
          RGT  CA 121-44-8 Et3N

     PRO  CL 396725-68-1

RX(34)    RCT  CL 396725-68-1, AT 191103-10-3

        STAGE(1)
          CAT  64-19-7 AcOH
          SOL  75-09-2 CH2Cl2

        STAGE(2)
          RGT  CK 16940-66-2 NaBH4

     PRO  BH 396725-70-5
     NTE  in-situ generated reactant


RX(202) OF 350 COMPOSED OF RX(35), RX(36), RX(37), RX(38)
RX(202)    CO  +  CF  +  AT  ===>  BM
```



CO



CF



BM
YIELD 4%

RX(35) RCT CO 4869-59-4, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CP 323191-90-8
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(36) RCT CP 323191-90-8

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CQ 396725-71-6

RX(37) RCT CQ 396725-71-6

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)

RGT CA 121-44-8 Et3N

PRO CR 396725-72-7

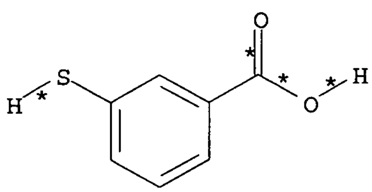
RX(38) RCT CR 396725-72-7, AT **191103-10-3**

STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

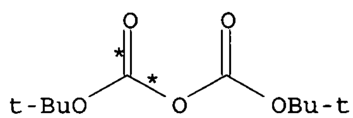
STAGE(2)
RGT CK 16940-66-2 NaBH4

PRO BM **396725-73-8**
NTE in-situ generated reactant

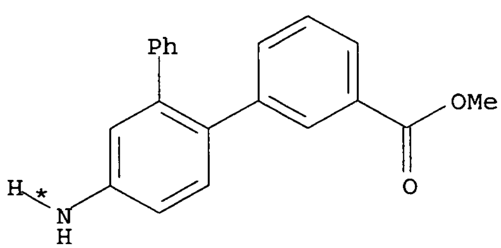
RX(203) OF 350 COMPOSED OF RX(35), RX(36), RX(37), RX(39)
RX(203) CO + **CF** + **AQ** ===> **BO**



CO

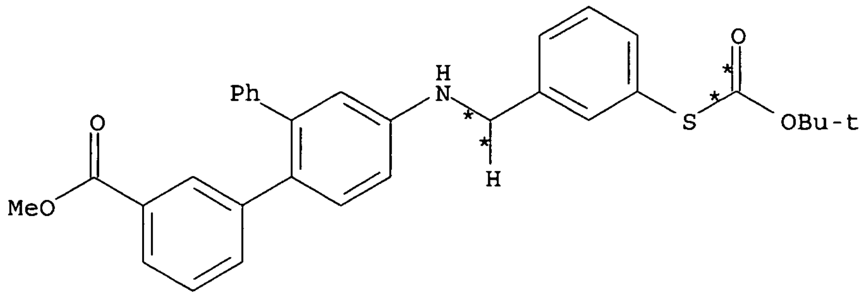


CF



AQ

4
STEPS
→



BO
YIELD 50%

RX(35) RCT CO 4869-59-4, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CP 323191-90-8
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(36) RCT CP 323191-90-8

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CQ 396725-71-6

RX(37) RCT CQ 396725-71-6

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CR 396725-72-7

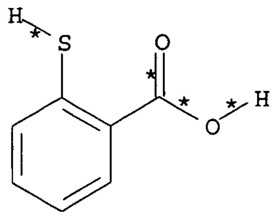
RX(39) RCT CR 396725-72-7, AQ **396725-50-1**

STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

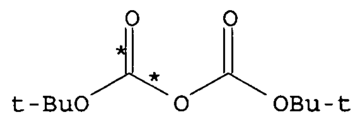
STAGE(2)
RGT CK 16940-66-2 NaBH4

PRO BO **396725-74-9**
NTE in-situ generated reactant

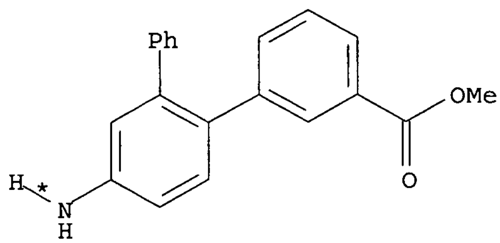
RX(212) OF 350 COMPOSED OF RX(40), RX(41), RX(42), RX(43)
RX(212) CS + **CF** + **AQ** ===> **BU**



CS

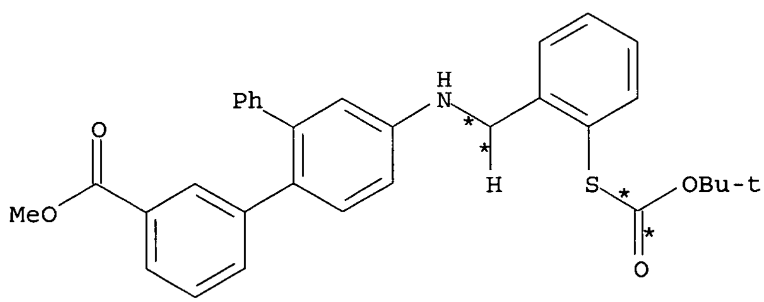


CF



AQ

4
STEPS
——>



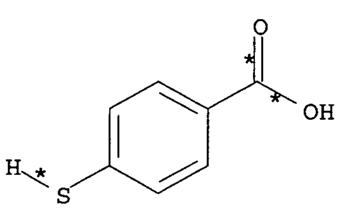
BU
YIELD 64%

RX(40) RCT CS 147-93-3, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CT 396725-75-0
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

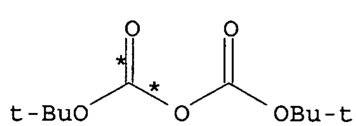
RX(41) RCT CT 396725-75-0

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

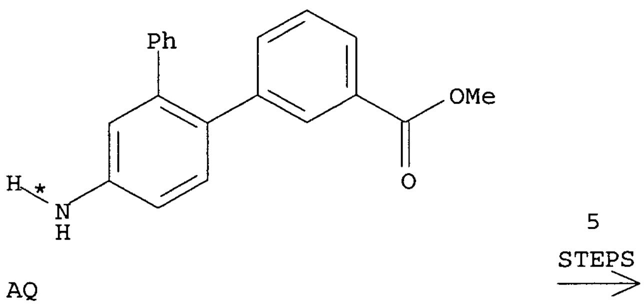
```
          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

          STAGE(3)
             RGT  O 7647-01-0 HCl
             SOL  7732-18-5 Water

        PRO  CU 396725-76-1

RX(42)  RCT  CU 396725-76-1

          STAGE(1)
             RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CA 121-44-8 Et3N

        PRO  CV 396725-77-2

RX(43)  RCT  CV 396725-77-2, AQ 396725-50-1

          STAGE(1)
             CAT  64-19-7 AcOH
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

        PRO  BU 396725-78-3
        NTE  in-situ generated reactant


RX(330) OF 350 COMPOSED OF RX(30), RX(31), RX(32), RX(33), RX(21)
RX(330)   CE  +  CF  +  AQ  ===>  BL
```



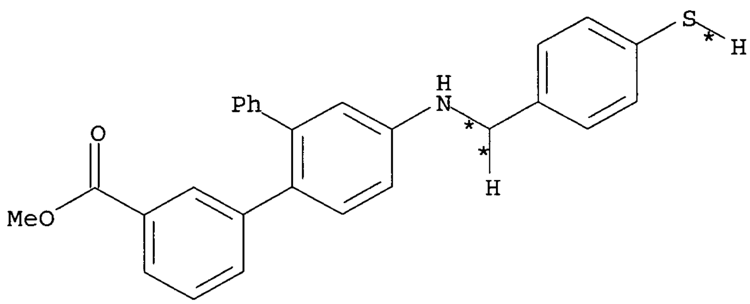
CE



CF



AQ



BL
YIELD 100%

RX(30) RCT CE 1074-36-8, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CG 396725-66-9
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(31) RCT CG 396725-66-9

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CI 396725-67-0

RX(32) RCT CI 396725-67-0

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CL 396725-68-1

RX(33) RCT CL 396725-68-1, AQ **396725-50-1**

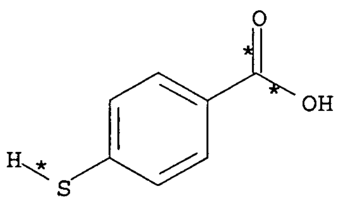
STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CK 16940-66-2 NaBH4

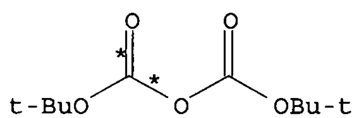
PRO BK 396725-69-2
NTE in-situ generated reactant

RX(21) RCT BK 396725-69-2
RGT B 76-05-1 F3CCO2H
PRO BL **396725-60-3**
SOL 75-09-2 CH2Cl2

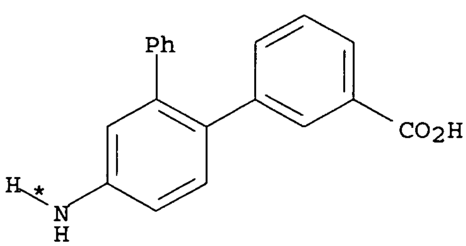
RX(331) OF 350 COMPOSED OF RX(30), RX(31), RX(32), RX(34), RX(20)
RX(331) CE + **CF** + **AT** ===> **BI**



CE

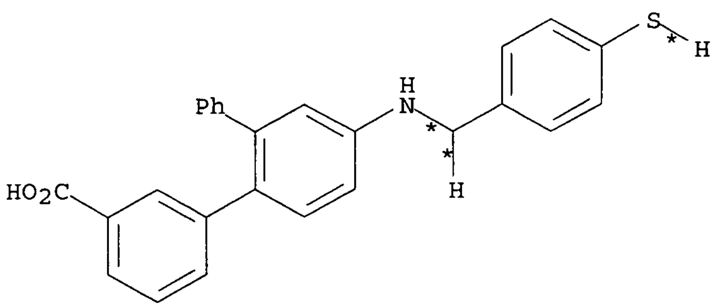


CF



AT

5 STEPS
$\longrightarrow$



BI
YIELD 87%

RX(30) RCT CE 1074-36-8, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CG 396725-66-9
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(31) RCT CG 396725-66-9

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CI 396725-67-0

RX(32) RCT CI 396725-67-0

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CL 396725-68-1

RX(34) RCT CL 396725-68-1, AT **191103-10-3**

STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CK 16940-66-2 NaBH4

PRO BH 396725-70-5

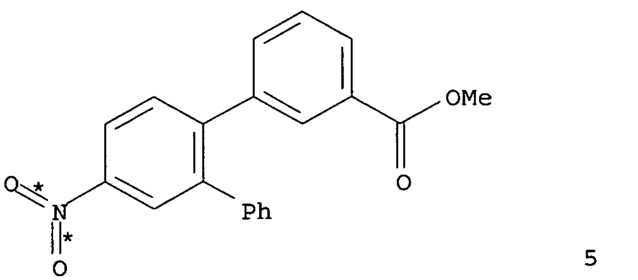
NTE in-situ generated reactant

RX(20) RCT BH 396725-70-5
RGT B 76-05-1 F3CCO2H
PRO BI **396725-59-0**
SOL 75-09-2 CH2Cl2

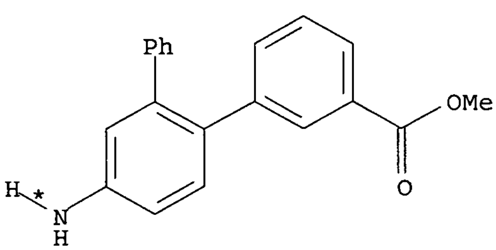
RX(332) OF 350 COMPOSED OF REACTION SEQUENCE RX(10), RX(33), RX(21)
AND REACTION SEQUENCE RX(30), RX(31), RX(32), RX(33), RX(21)

...AL ===> AQ...

...CE + **CF** + **AQ** ===> **BL**

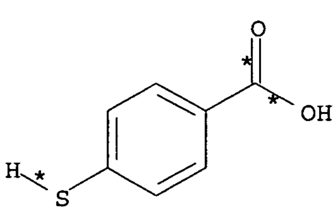


AL

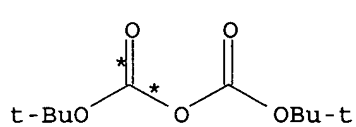


AQ

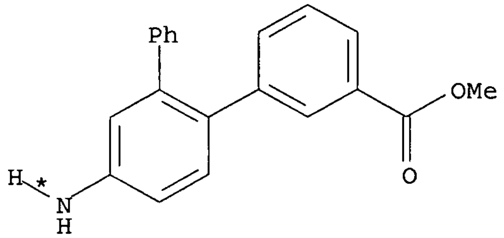
START NEXT REACTION SEQUENCE



CE

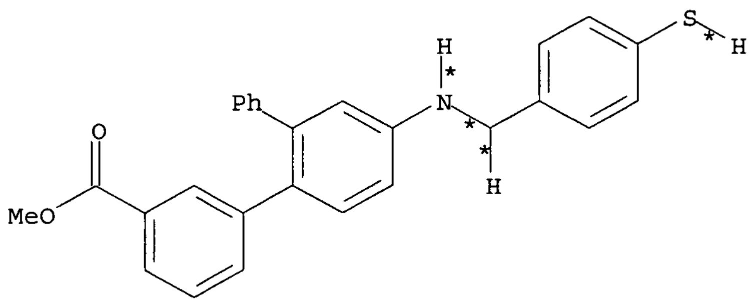


CF



AQ

5 STEPS
$\longrightarrow$



BL
YIELD 100%

```
RX(10)     RCT  AL 323191-84-0
           RGT  AR 7772-99-8 SnCl2
           PRO  AQ 396725-50-1
           SOL  141-78-6 AcOEt

RX(30)     RCT  CE 1074-36-8, CF 24424-99-5
           RGT  CA 121-44-8 Et3N
           PRO  CG 396725-66-9
           CAT  1122-58-3 4-DMAP
           SOL  109-99-9 THF

RX(31)     RCT  CG 396725-66-9

             STAGE(1)
                RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
                SOL  109-99-9 THF

             STAGE(2)
                RGT  CK 16940-66-2 NaBH4

             STAGE(3)
                RGT  O 7647-01-0 HCl
                SOL  7732-18-5 Water

           PRO  CI 396725-67-0

RX(32)     RCT  CI 396725-67-0
```

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CL 396725-68-1

RX(33) RCT CL 396725-68-1, AQ **396725-50-1**

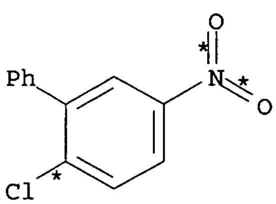
STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CK 16940-66-2 NaBH4

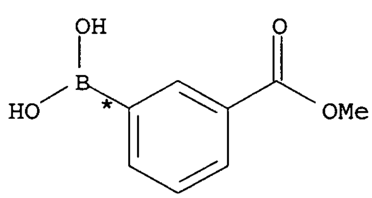
PRO BK 396725-69-2
NTE in-situ generated reactant

RX(21) RCT BK 396725-69-2
RGT B 76-05-1 F3CCO2H
PRO BL **396725-60-3**
SOL 75-09-2 CH2Cl2

RX(333) OF 350 COMPOSED OF REACTION SEQUENCE RX(9), RX(10), RX(33), RX(21)
AND REACTION SEQUENCE RX(30), RX(31), RX(32), RX(33), RX(21)
...AJ + AK ===> AQ...
...CE + **CF** + **AQ** ===> **BL**

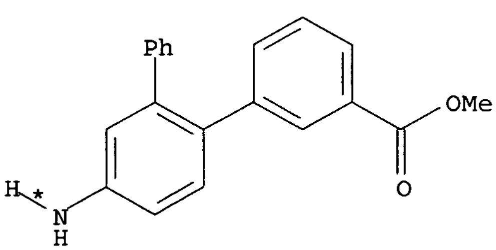


AJ



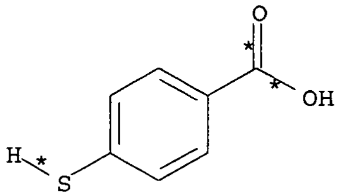
AK

5
STEPS

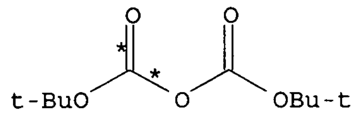


AQ

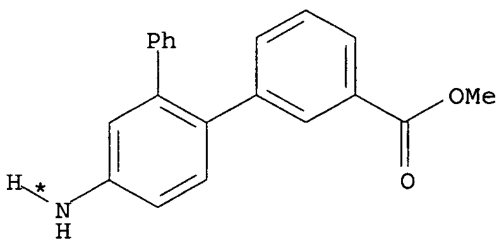
START NEXT REACTION SEQUENCE



CE

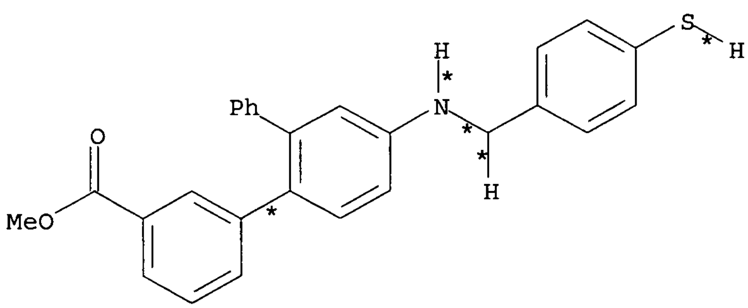


CF



AQ

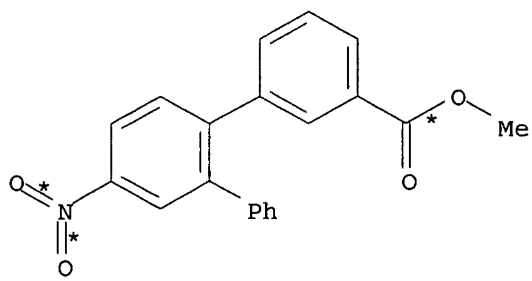
5 STEPS →



BL
YIELD 100%

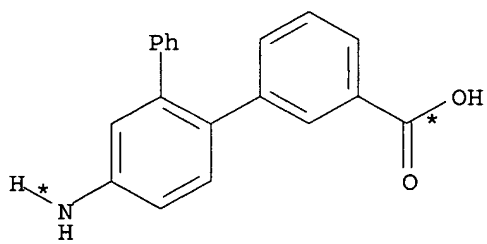
RX(9)	RCT	AJ 29608-76-2, AK 99769-19-4
	RGT	AM 7778-53-2 K3PO4, AN 247940-06-3 Phosphine, [1,1'-biphenyl]-2-yldicyclohexyl-
	PRO	AL 323191-84-0
	CAT	3375-31-3 Pd(OAc)2
	SOL	108-88-3 PhMe
RX(10)	RCT	AL 323191-84-0
	RGT	AR 7772-99-8 SnCl2
	PRO	AQ 396725-50-1
	SOL	141-78-6 AcOEt
RX(30)	RCT	CE 1074-36-8, CF **24424-99-5**
	RGT	CA 121-44-8 Et3N
	PRO	CG 396725-66-9

```
        CAT   1122-58-3 4-DMAP
        SOL   109-99-9 THF

RX(31)  RCT   CG 396725-66-9

          STAGE(1)
             RGT   CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
             SOL   109-99-9 THF

          STAGE(2)
             RGT   CK 16940-66-2 NaBH4

          STAGE(3)
             RGT   O 7647-01-0 HCl
             SOL   7732-18-5 Water

        PRO   CI 396725-67-0

RX(32)  RCT   CI 396725-67-0

          STAGE(1)
             RGT   CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
             SOL   75-09-2 CH2Cl2

          STAGE(2)
             RGT   CA 121-44-8 Et3N

        PRO   CL 396725-68-1

RX(33)  RCT   CL 396725-68-1, AQ 396725-50-1

          STAGE(1)
             CAT   64-19-7 AcOH
             SOL   75-09-2 CH2Cl2

          STAGE(2)
             RGT   CK 16940-66-2 NaBH4

        PRO   BK 396725-69-2
        NTE   in-situ generated reactant

RX(21)  RCT   BK 396725-69-2
        RGT   B 76-05-1 F3CCO2H
        PRO   BL 396725-60-3
        SOL   75-09-2 CH2Cl2


RX(334) OF 350 COMPOSED OF REACTION SEQUENCE RX(11), RX(34), RX(20)
               AND REACTION SEQUENCE RX(30), RX(31), RX(32), RX(34), RX(20)
...AL  ===>  AT...
...CE  +  CF  +  AT  ===>  BI
```



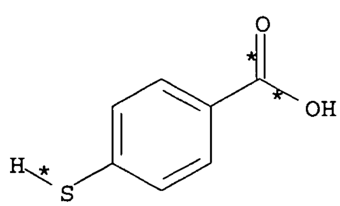
AL

5
STEPS
→

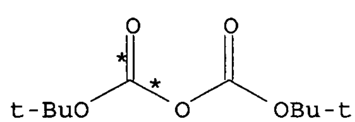


AT

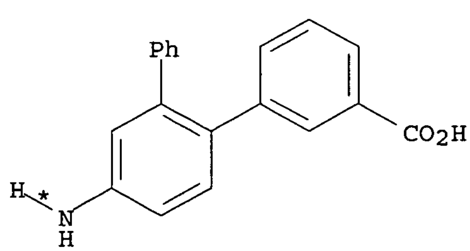
START NEXT REACTION SEQUENCE



CE

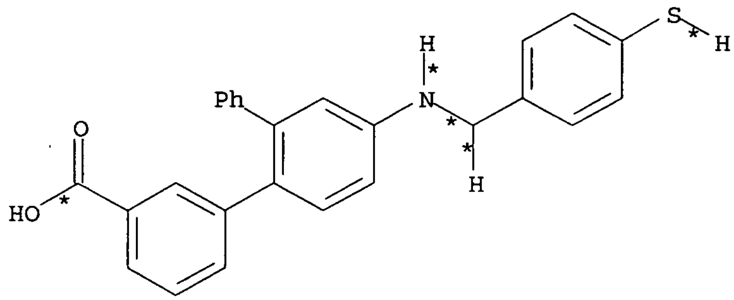


CF



AT

5
STEPS
→



BI
YIELD 87%

RX(11) RCT AL 323191-84-0

STAGE(1)
RGT AU 1310-65-2 LiOH
SOL 109-99-9 THF, 7732-18-5 Water

STAGE(2)
RGT AR 7772-99-8 SnCl2
SOL 141-78-6 AcOEt

PRO AT 191103-10-3

RX(30) RCT CE 1074-36-8, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CG 396725-66-9
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(31) RCT CG 396725-66-9

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CI 396725-67-0

RX(32) RCT CI 396725-67-0

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CL 396725-68-1

RX(34) RCT CL 396725-68-1, AT **191103-10-3**

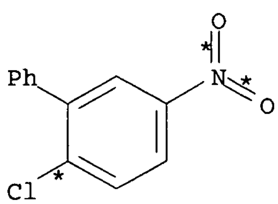
STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CK 16940-66-2 NaBH4

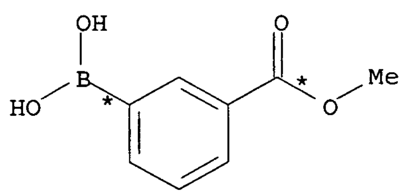
PRO BH 396725-70-5
NTE in-situ generated reactant

RX(20) RCT BH 396725-70-5
RGT B 76-05-1 F3CCO2H
PRO BI **396725-59-0**
SOL 75-09-2 CH2Cl2

RX(335) OF 350 COMPOSED OF REACTION SEQUENCE RX(9), RX(11), RX(34), RX(20)
AND REACTION SEQUENCE RX(30), RX(31), RX(32), RX(34), RX(20)
...AJ + AK ===> AT...
...CE + **CF** + **AT** ===> **BI**

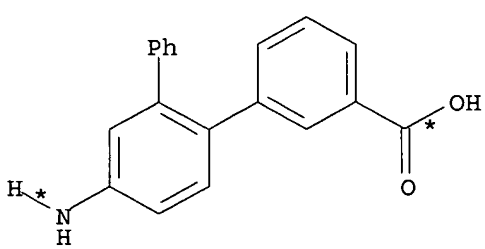


AJ



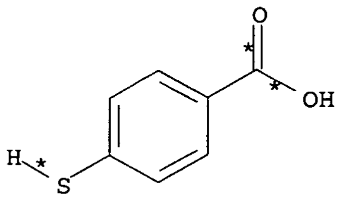
AK

5
STEPS
→

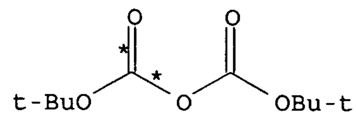


AT

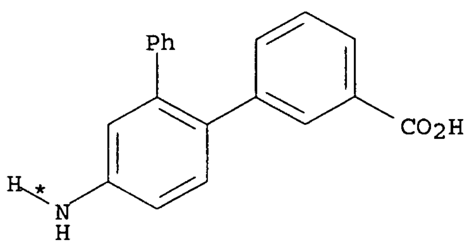
START NEXT REACTION SEQUENCE



CE

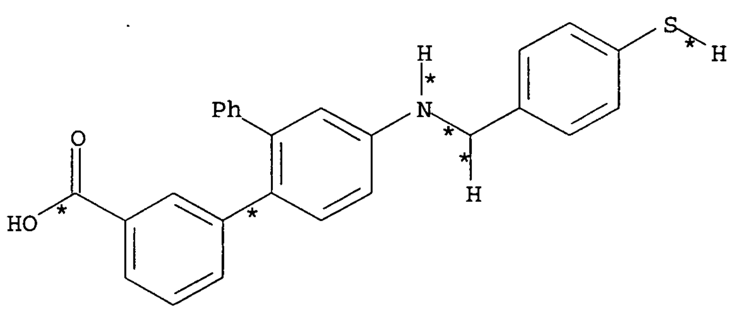


CF



AT

5
STEPS
→



BI
YIELD 87%

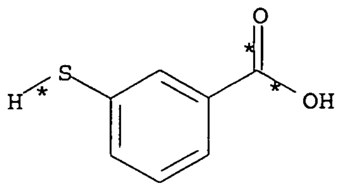
RX(9) RCT AJ 29608-76-2, AK 99769-19-4
RGT AM 7778-53-2 K3PO4, AN 247940-06-3 Phosphine,
[1,1'-biphenyl]-2-yldicyclohexyl-
PRO AL 323191-84-0
CAT 3375-31-3 Pd(OAc)2
SOL 108-88-3 PhMe

RX(11) RCT AL 323191-84-0

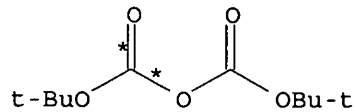
STAGE(1)
RGT AU 1310-65-2 LiOH
SOL 109-99-9 THF, 7732-18-5 Water

STAGE(2)

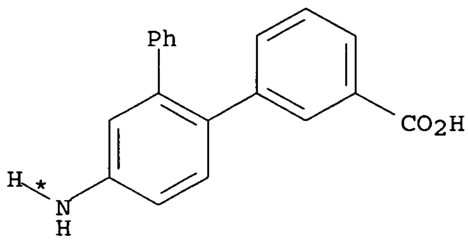
```
            RGT  AR 7772-99-8 SnCl2
            SOL  141-78-6 AcOEt

       PRO  AT 191103-10-3

RX(30) RCT  CE 1074-36-8, CF 24424-99-5
       RGT  CA 121-44-8 Et3N
       PRO  CG 396725-66-9
       CAT  1122-58-3 4-DMAP
       SOL  109-99-9 THF

RX(31) RCT  CG 396725-66-9

         STAGE(1)
            RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
            SOL  109-99-9 THF

         STAGE(2)
            RGT  CK 16940-66-2 NaBH4

         STAGE(3)
            RGT  O 7647-01-0 HCl
            SOL  7732-18-5 Water

       PRO  CI 396725-67-0

RX(32) RCT  CI 396725-67-0

         STAGE(1)
            RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
            SOL  75-09-2 CH2Cl2

         STAGE(2)
            RGT  CA 121-44-8 Et3N

       PRO  CL 396725-68-1

RX(34) RCT  CL 396725-68-1, AT 191103-10-3

         STAGE(1)
            CAT  64-19-7 AcOH
            SOL  75-09-2 CH2Cl2

         STAGE(2)
            RGT  CK 16940-66-2 NaBH4

       PRO  BH 396725-70-5
       NTE  in-situ generated reactant

RX(20) RCT  BH 396725-70-5
       RGT  B 76-05-1 F3CCO2H
       PRO  BI 396725-59-0
       SOL  75-09-2 CH2Cl2


RX(336) OF 350 COMPOSED OF RX(35), RX(36), RX(37), RX(38), RX(22)
RX(336)    CO  +  CF  +  AT  ===>  BN
```



CO

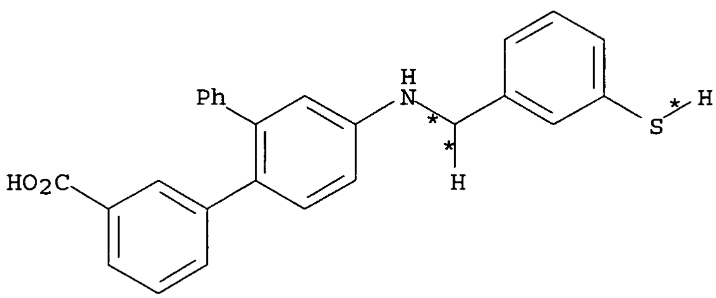


CF



AT

5
STEPS
→



BN
YIELD 88%

RX(35) RCT CO 4869-59-4, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CP 323191-90-8
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

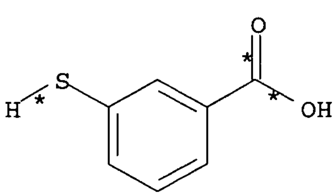
RX(36) RCT CP 323191-90-8

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

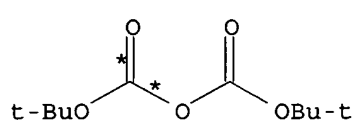
STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)

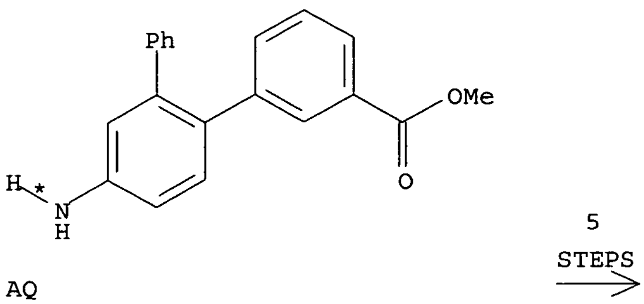
```
          RGT  O 7647-01-0 HCl
          SOL  7732-18-5 Water

     PRO  CQ 396725-71-6

RX(37)     RCT  CQ 396725-71-6

       STAGE(1)
          RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
          SOL  75-09-2 CH2Cl2

       STAGE(2)
          RGT  CA 121-44-8 Et3N

     PRO  CR 396725-72-7

RX(38)     RCT  CR 396725-72-7, AT 191103-10-3

       STAGE(1)
          CAT  64-19-7 AcOH
          SOL  75-09-2 CH2Cl2

       STAGE(2)
          RGT  CK 16940-66-2 NaBH4

     PRO  BM 396725-73-8
     NTE  in-situ generated reactant

RX(22)     RCT  BM 396725-73-8
     RGT  B 76-05-1 F3CCO2H
     PRO  BN 396725-61-4
     SOL  75-09-2 CH2Cl2


RX(337) OF 350 COMPOSED OF RX(35), RX(36), RX(37), RX(39), RX(23)
RX(337)    CO  +  CF  +  AQ  ===>  BP
```



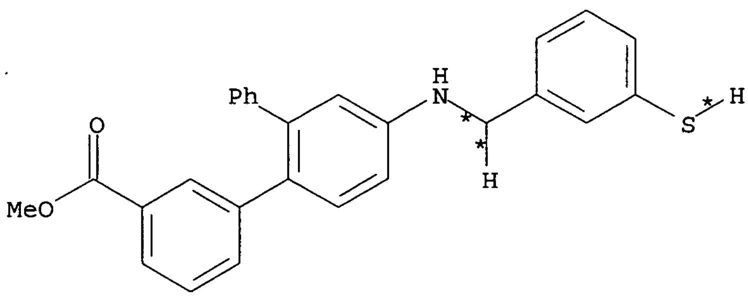
CO



CF



AQ



BP
YIELD 100%

RX(35) RCT CO 4869-59-4, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CP 323191-90-8
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(36) RCT CP 323191-90-8

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CQ 396725-71-6

RX(37) RCT CQ 396725-71-6

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)

RGT CA 121-44-8 Et3N

PRO CR 396725-72-7

RX(39) RCT CR 396725-72-7, AQ **396725-50-1**

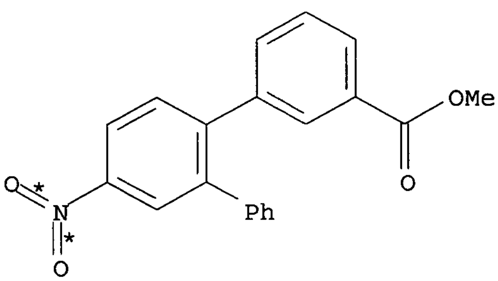
STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CK 16940-66-2 NaBH4

PRO BO 396725-74-9
NTE in-situ generated reactant

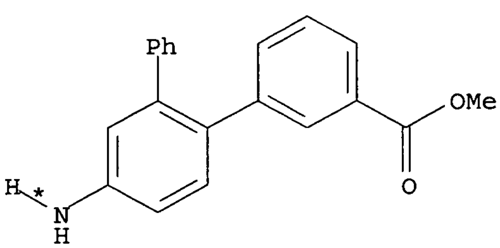
RX(23) RCT BO 396725-74-9
RGT B 76-05-1 F3CCO2H
PRO BP **396725-62-5**
SOL 75-09-2 CH2Cl2

RX(338) OF 350 COMPOSED OF REACTION SEQUENCE RX(10), RX(39), RX(23)
AND REACTION SEQUENCE RX(35), RX(36), RX(37), RX(39), RX(23)
...AL ===> AQ...
...CO + **CF** + **AQ** ===> **BP**



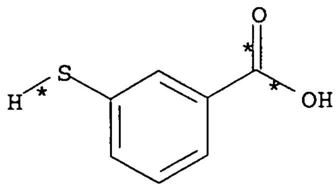
AL

5
STEPS

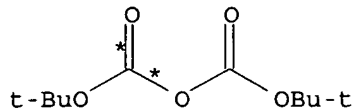


AQ

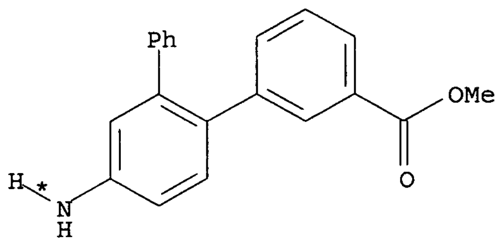
START NEXT REACTION SEQUENCE



CO

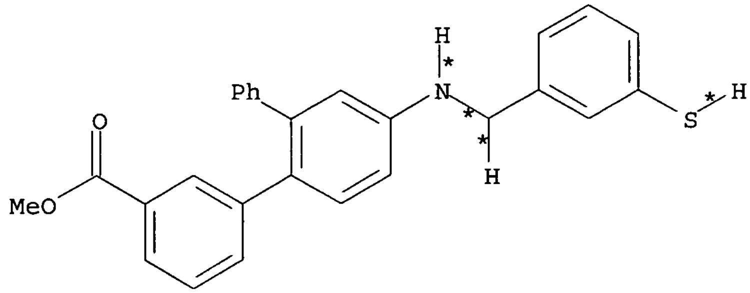


CF



AQ

5
STEPS
$\longrightarrow$



BP
YIELD 100%

```
RX(10)     RCT  AL 323191-84-0
           RGT  AR 7772-99-8 SnCl2
           PRO  AQ 396725-50-1
           SOL  141-78-6 AcOEt

RX(35)     RCT  CO 4869-59-4, CF 24424-99-5
           RGT  CA 121-44-8 Et3N
           PRO  CP 323191-90-8
           CAT  1122-58-3 4-DMAP
           SOL  109-99-9 THF

RX(36)     RCT  CP 323191-90-8

             STAGE(1)
                RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
```

SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CQ 396725-71-6

RX(37) RCT CQ 396725-71-6

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CR 396725-72-7

RX(39) RCT CR 396725-72-7, AQ **396725-50-1**

STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

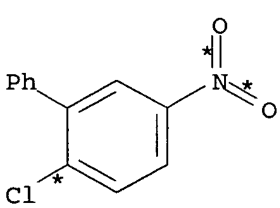
STAGE(2)
RGT CK 16940-66-2 NaBH4

PRO BO 396725-74-9
NTE in-situ generated reactant

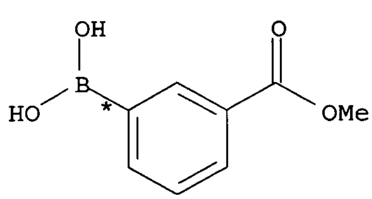
RX(23) RCT BO 396725-74-9
RGT B 76-05-1 F3CCO2H
PRO BP **396725-62-5**
SOL 75-09-2 CH2Cl2

RX(339) OF 350 COMPOSED OF REACTION SEQUENCE RX(9), RX(10), RX(39), RX(23)
AND REACTION SEQUENCE RX(35), RX(36), RX(37), RX(39), RX(23)

...AJ + AK ===> AQ...
...CO + **CF** + **AQ** ===> **BP**

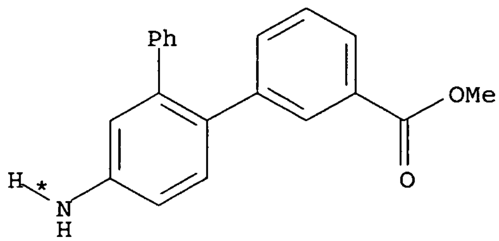


AJ



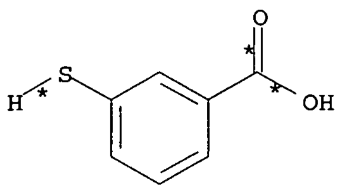
AK

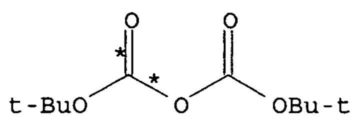
5
STEPS
$\longrightarrow$



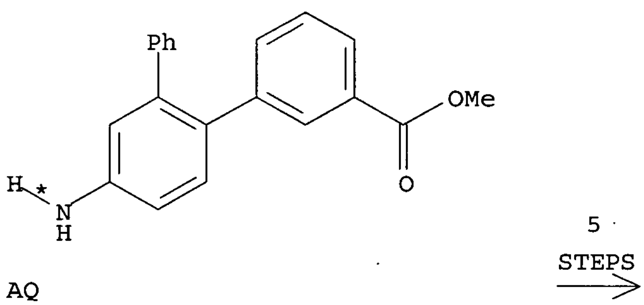
AQ

START NEXT REACTION SEQUENCE

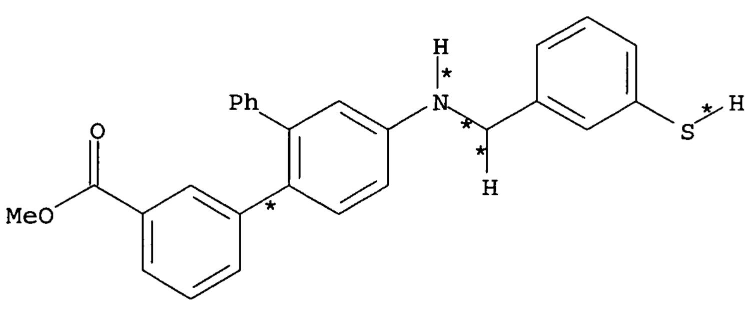




CO CF



AQ



BP
YIELD 100%

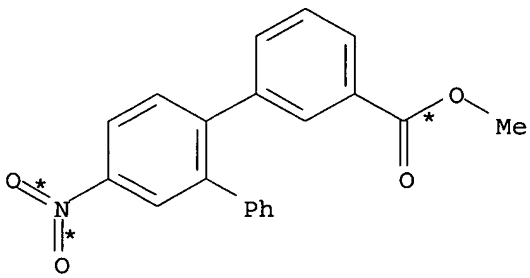
RX(9) RCT AJ 29608-76-2, AK 99769-19-4

```
        RGT  AM 7778-53-2 K3PO4, AN 247940-06-3 Phosphine,
             [1,1'-biphenyl]-2-yldicyclohexyl-
        PRO  AL 323191-84-0
        CAT  3375-31-3 Pd(OAc)2
        SOL  108-88-3 PhMe

RX(10)  RCT  AL 323191-84-0
        RGT  AR 7772-99-8 SnCl2
        PRO  AQ 396725-50-1
        SOL  141-78-6 AcOEt

RX(35)  RCT  CO 4869-59-4, CF 24424-99-5
        RGT  CA 121-44-8 Et3N
        PRO  CP 323191-90-8
        CAT  1122-58-3 4-DMAP
        SOL  109-99-9 THF

RX(36)  RCT  CP 323191-90-8

          STAGE(1)
             RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
             SOL  109-99-9 THF

          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

          STAGE(3)
             RGT  O 7647-01-0 HCl
             SOL  7732-18-5 Water

        PRO  CQ 396725-71-6

RX(37)  RCT  CQ 396725-71-6

          STAGE(1)
             RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CA 121-44-8 Et3N

        PRO  CR 396725-72-7

RX(39)  RCT  CR 396725-72-7, AQ 396725-50-1

          STAGE(1)
             CAT  64-19-7 AcOH
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

        PRO  BO 396725-74-9
        NTE  in-situ generated reactant

RX(23)  RCT  BO 396725-74-9
        RGT  B 76-05-1 F3CCO2H
        PRO  BP 396725-62-5
        SOL  75-09-2 CH2Cl2
```

RX(340) OF 350 COMPOSED OF REACTION SEQUENCE RX(11), RX(38), RX(22)
AND REACTION SEQUENCE RX(35), RX(36), RX(37), RX(38), RX(22)

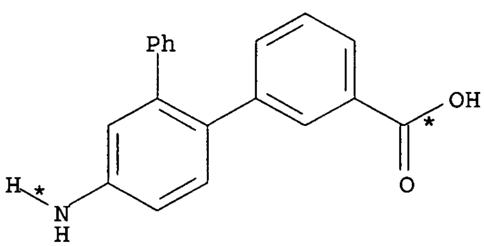
...AL ===> AT...

...CO + **CF** + **AT** ===> **BN**



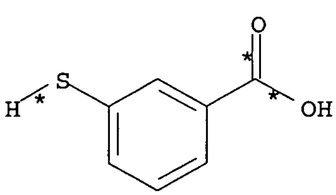
AL

5
STEPS
→

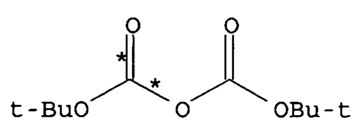


AT

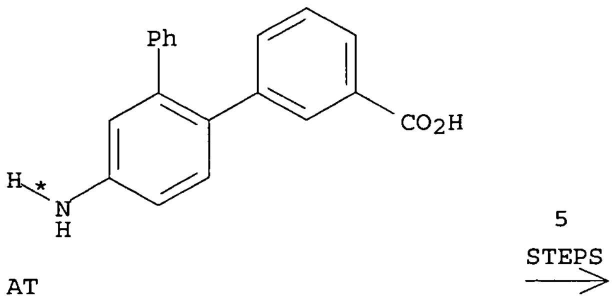
START NEXT REACTION SEQUENCE



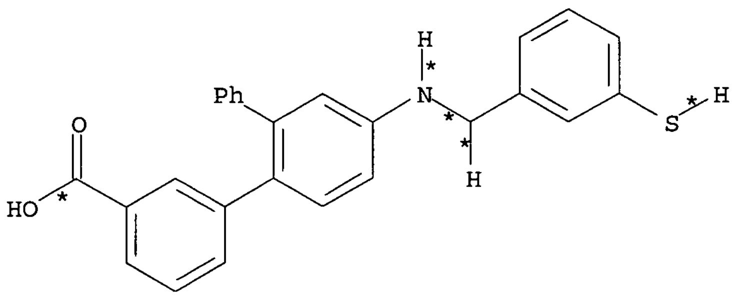
CO



CF



AT



BN
YIELD 88%

RX(11) RCT AL 323191-84-0

STAGE(1)
RGT AU 1310-65-2 LiOH
SOL 109-99-9 THF, 7732-18-5 Water

STAGE(2)
RGT AR 7772-99-8 SnCl2
SOL 141-78-6 AcOEt

PRO AT 191103-10-3

RX(35) RCT CO 4869-59-4, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CP 323191-90-8
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

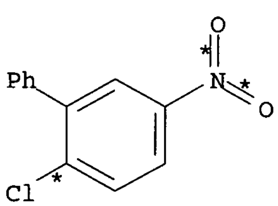
RX(36) RCT CP 323191-90-8

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

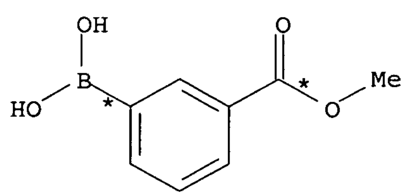
STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)

```
            RGT  O 7647-01-0 HCl
            SOL  7732-18-5 Water

       PRO  CQ 396725-71-6

RX(37)  RCT  CQ 396725-71-6

          STAGE(1)
            RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
            SOL  75-09-2 CH2Cl2

          STAGE(2)
            RGT  CA 121-44-8 Et3N

       PRO  CR 396725-72-7

RX(38)  RCT  CR 396725-72-7, AT 191103-10-3

          STAGE(1)
            CAT  64-19-7 AcOH
            SOL  75-09-2 CH2Cl2

          STAGE(2)
            RGT  CK 16940-66-2 NaBH4

       PRO  BM 396725-73-8
       NTE  in-situ generated reactant

RX(22)  RCT  BM 396725-73-8
        RGT  B 76-05-1 F3CCO2H
        PRO  BN 396725-61-4
        SOL  75-09-2 CH2Cl2


RX(341) OF 350 COMPOSED OF REACTION SEQUENCE RX(9), RX(11), RX(38), RX(22)
               AND REACTION SEQUENCE RX(35), RX(36), RX(37), RX(38), RX(22)
...AJ  +  AK  ===>  AT...
...CO  +  CF  +  AT  ===>  BN
```



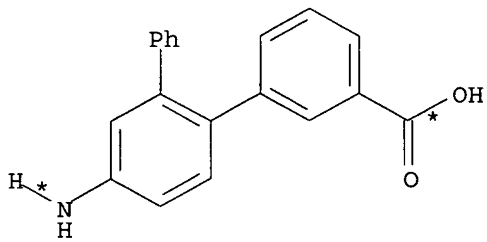
AJ



AK

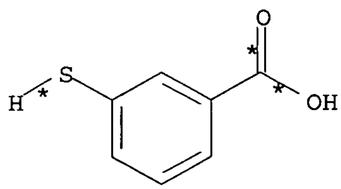
5

STEPS

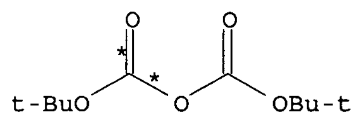


AT

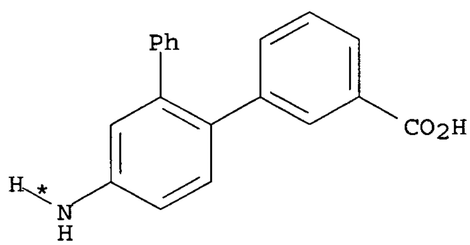
START NEXT REACTION SEQUENCE



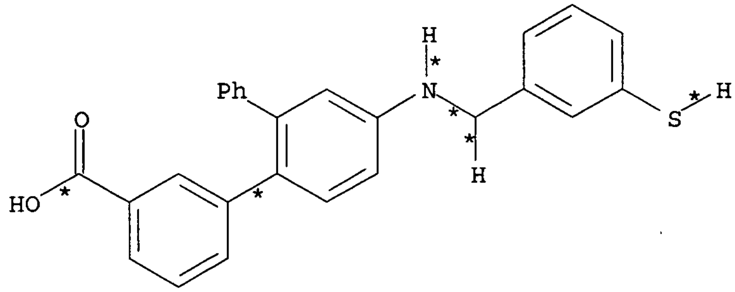
CO



CF



AT



BN
YIELD 88%

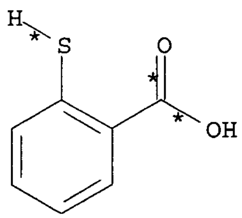
RX(9) RCT AJ 29608-76-2, AK 99769-19-4

```
        RGT  AM 7778-53-2 K3PO4, AN 247940-06-3 Phosphine,
             [1,1'-biphenyl]-2-yldicyclohexyl-
        PRO  AL 323191-84-0
        CAT  3375-31-3 Pd(OAc)2
        SOL  108-88-3 PhMe

RX(11)  RCT  AL 323191-84-0

          STAGE(1)
             RGT  AU 1310-65-2 LiOH
             SOL  109-99-9 THF, 7732-18-5 Water

          STAGE(2)
             RGT  AR 7772-99-8 SnCl2
             SOL  141-78-6 AcOEt

        PRO  AT 191103-10-3

RX(35)  RCT  CO 4869-59-4, CF 24424-99-5
        RGT  CA 121-44-8 Et3N
        PRO  CP 323191-90-8
        CAT  1122-58-3 4-DMAP
        SOL  109-99-9 THF

RX(36)  RCT  CP 323191-90-8

          STAGE(1)
             RGT  CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
             SOL  109-99-9 THF

          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

          STAGE(3)
             RGT  O 7647-01-0 HCl
             SOL  7732-18-5 Water

        PRO  CQ 396725-71-6

RX(37)  RCT  CQ 396725-71-6

          STAGE(1)
             RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CA 121-44-8 Et3N

        PRO  CR 396725-72-7

RX(38)  RCT  CR 396725-72-7, AT 191103-10-3

          STAGE(1)
             CAT  64-19-7 AcOH
             SOL  75-09-2 CH2Cl2

          STAGE(2)
             RGT  CK 16940-66-2 NaBH4

        PRO  BM 396725-73-8
```

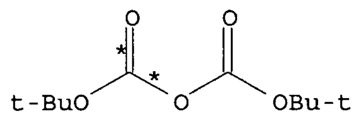
NTE in-situ generated reactant

RX(22) RCT BM 396725-73-8
RGT B 76-05-1 F3CCO2H
PRO BN **396725-61-4**
SOL 75-09-2 CH2Cl2

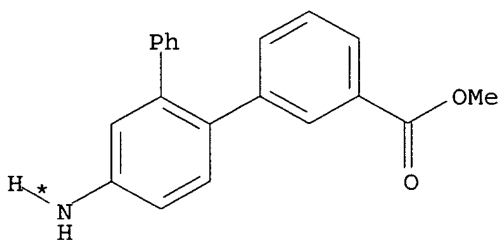
RX(342) OF 350 COMPOSED OF RX(40), RX(41), RX(42), RX(43), RX(26)
RX(342) CS + **CF** + **AQ** ===> **BV**



CS

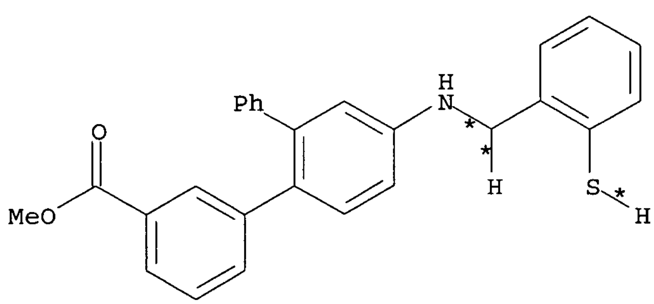


CF



AQ

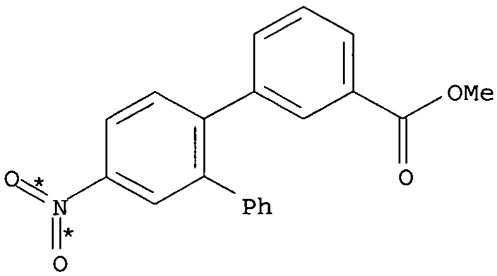
5
STEPS



BV
YIELD 100%

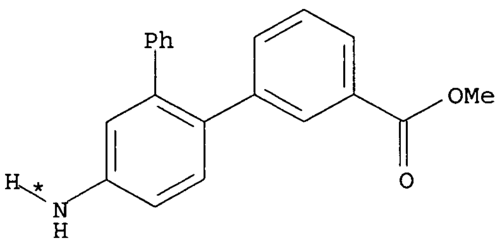
RX(40) RCT CS 147-93-3, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CT 396725-75-0

```
        CAT   1122-58-3 4-DMAP
        SOL   109-99-9 THF

RX(41)  RCT   CT 396725-75-0

          STAGE(1)
             RGT   CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
             SOL   109-99-9 THF

          STAGE(2)
             RGT   CK 16940-66-2 NaBH4

          STAGE(3)
             RGT   O 7647-01-0 HCl
             SOL   7732-18-5 Water

        PRO   CU 396725-76-1

RX(42)  RCT   CU 396725-76-1

          STAGE(1)
             RGT   CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
             SOL   75-09-2 CH2Cl2

          STAGE(2)
             RGT   CA 121-44-8 Et3N

        PRO   CV 396725-77-2

RX(43)  RCT   CV 396725-77-2, AQ 396725-50-1

          STAGE(1)
             CAT   64-19-7 AcOH
             SOL   75-09-2 CH2Cl2

          STAGE(2)
             RGT   CK 16940-66-2 NaBH4

        PRO   BU 396725-78-3
        NTE   in-situ generated reactant

RX(26)  RCT   BU 396725-78-3
        RGT   B 76-05-1 F3CCO2H
        PRO   BV 396725-64-7
        SOL   75-09-2 CH2Cl2


RX(343) OF 350 COMPOSED OF REACTION SEQUENCE RX(10), RX(43), RX(26)
               AND REACTION SEQUENCE RX(40), RX(41), RX(42), RX(43), RX(26)
...AL  ===>  AQ...
...CS  +  CF  +  AQ  ===>  BV
```



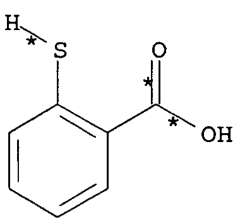
AL

5
STEPS
→

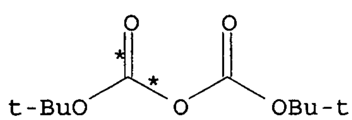


AQ

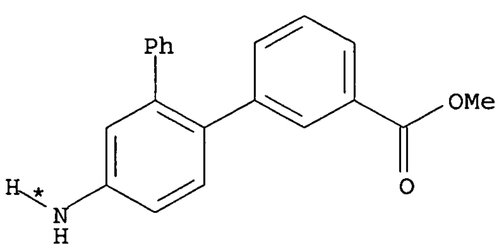
START NEXT REACTION SEQUENCE



CS

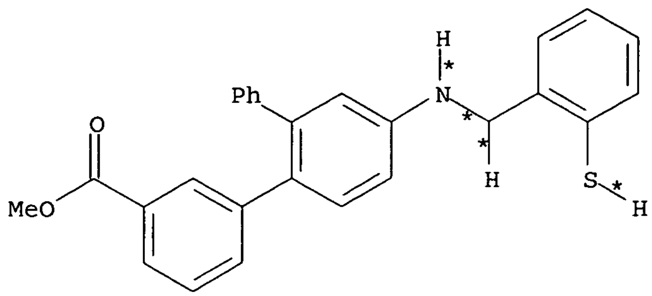


CF



AQ

5
STEPS
→



BV
YIELD 100%

RX(10) RCT AL 323191-84-0
RGT AR 7772-99-8 SnCl2
PRO AQ 396725-50-1
SOL 141-78-6 AcOEt

RX(40) RCT CS 147-93-3, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CT 396725-75-0
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(41) RCT CT 396725-75-0

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)
RGT O 7647-01-0 HCl
SOL 7732-18-5 Water

PRO CU 396725-76-1

RX(42) RCT CU 396725-76-1

STAGE(1)
RGT CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
SOL 75-09-2 CH2Cl2

STAGE(2)
RGT CA 121-44-8 Et3N

PRO CV 396725-77-2

RX(43) RCT CV 396725-77-2, AQ **396725-50-1**

STAGE(1)
CAT 64-19-7 AcOH
SOL 75-09-2 CH2Cl2

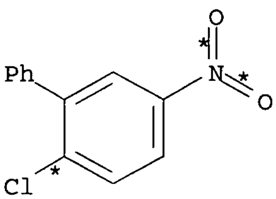
STAGE(2)

RGT CK 16940-66-2 NaBH4

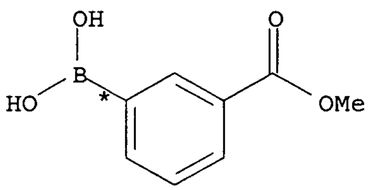
PRO BU 396725-78-3
NTE in-situ generated reactant

RX(26) RCT BU 396725-78-3
RGT B 76-05-1 F3CCO2H
PRO BV **396725-64-7**
SOL 75-09-2 CH2Cl2

RX(344) OF 350 COMPOSED OF REACTION SEQUENCE RX(9), RX(10), RX(43), RX(26)
AND REACTION SEQUENCE RX(40), RX(41), RX(42), RX(43), RX(26)
...AJ + AK ===> AQ...
...CS + **CF** + **AQ** ===> **BV**

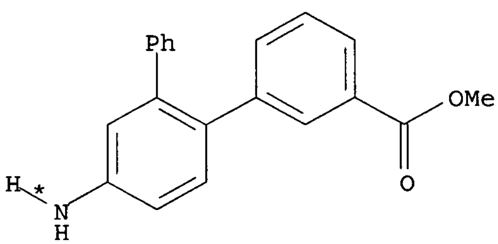


AJ



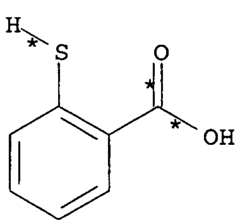
AK

5
STEPS

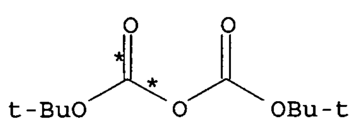


AQ

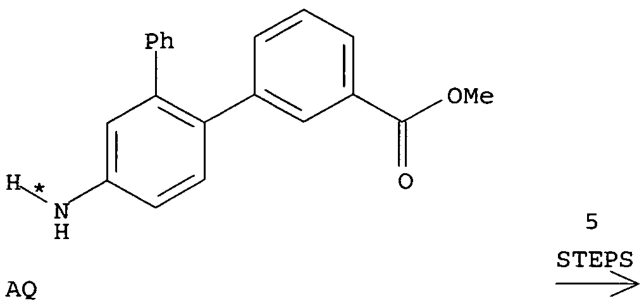
START NEXT REACTION SEQUENCE



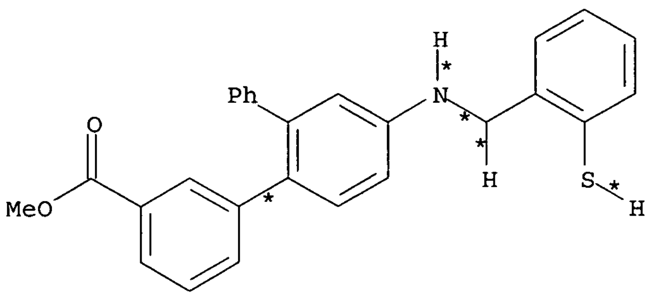
CS



CF



AQ



BV
YIELD 100%

RX(9) RCT AJ 29608-76-2, AK 99769-19-4
RGT AM 7778-53-2 K3PO4, AN 247940-06-3 Phosphine, [1,1'-biphenyl]-2-yldicyclohexyl-
PRO AL 323191-84-0
CAT 3375-31-3 Pd(OAc)2
SOL 108-88-3 PhMe

RX(10) RCT AL 323191-84-0
RGT AR 7772-99-8 SnCl2
PRO AQ 396725-50-1
SOL 141-78-6 AcOEt

RX(40) RCT CS 147-93-3, CF **24424-99-5**
RGT CA 121-44-8 Et3N
PRO CT 396725-75-0
CAT 1122-58-3 4-DMAP
SOL 109-99-9 THF

RX(41) RCT CT 396725-75-0

STAGE(1)
RGT CA 121-44-8 Et3N, CJ 541-41-3 ClCO2Et
SOL 109-99-9 THF

STAGE(2)
RGT CK 16940-66-2 NaBH4

STAGE(3)

```
            RGT  O 7647-01-0 HCl
            SOL  7732-18-5 Water

        PRO  CU 396725-76-1

RX(42)  RCT  CU 396725-76-1

          STAGE(1)
            RGT  CM 79-37-8 (COCl)2, CN 67-68-5 DMSO
            SOL  75-09-2 CH2Cl2

          STAGE(2)
            RGT  CA 121-44-8 Et3N

        PRO  CV 396725-77-2

RX(43)  RCT  CV 396725-77-2, AQ 396725-50-1

          STAGE(1)
            CAT  64-19-7 AcOH
            SOL  75-09-2 CH2Cl2

          STAGE(2)
            RGT  CK 16940-66-2 NaBH4

        PRO  BU 396725-78-3
        NTE  in-situ generated reactant

RX(26)  RCT  BU 396725-78-3
        RGT  B 76-05-1 F3CCO2H
        PRO  BV 396725-64-7
        SOL  75-09-2 CH2Cl2
```

L60 ANSWER 26 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 137:352757 CASREACT

TITLE: Catalytic Activity of Mesoporous Silica for Synthesis of Methyl N-Phenyl Carbamate from Dimethyl Carbonate and Aniline

AUTHOR(S): Katada, Naonobu; Fujinaga, Haruhisa; Nakamura, Yukinori; Okumura, Kazu; Nishigaki, Kyoichi; Niwa, Miki

CORPORATE SOURCE: Faculty of Engineering, Department of Materials Science, Tottori University, Tottori, 680-8552, Japan

SOURCE: Catalysis Letters (2002), 80(1-2), 47-51
CODEN: CALEER; ISSN: 1011-372X

PUBLISHER: Kluwer Academic/Plenum Publishers

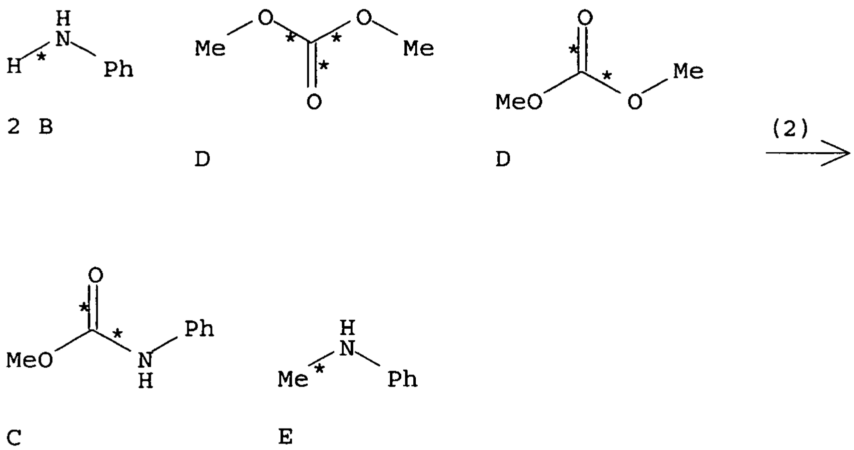
DOCUMENT TYPE: Journal

LANGUAGE: English

AB Mesoporous silica MCM-41, especially an Al-containing one, showed high catalytic activity for synthesis of phenylcarbamic acid Me ester from di-Me carbonate and aniline at 363-383 K. It was easily separated from the product solution, and the catalyst was able to be used repeatedly.

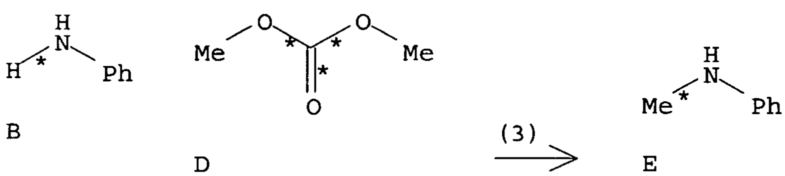
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(2) OF 6 2 **B** + 2 **D** ===> C + **E**



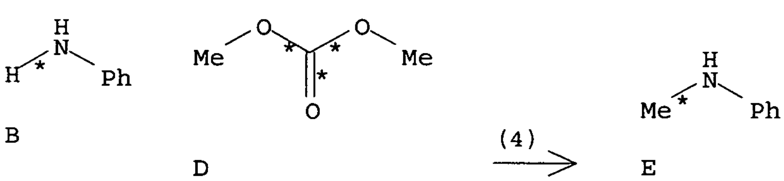
RX(2) RCT B **62-53-3**, D **616-38-6**
PRO C 2603-10-3, E **100-61-8**
CAT 7631-86-9D SiO2

RX(3) OF 6 **B** + **D** ===> **E**



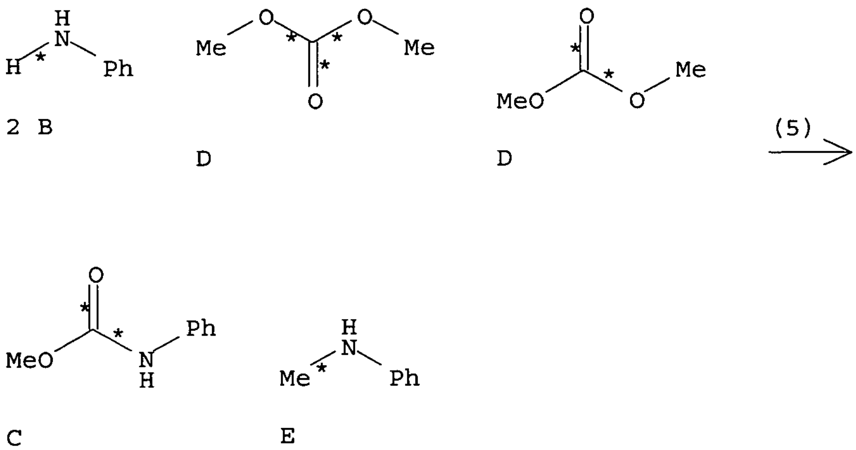
RX(3) RCT B **62-53-3**, D **616-38-6**
PRO E **100-61-8**
CAT 7631-86-9D SiO2

RX(4) OF 6 **B** + **D** ===> **E**



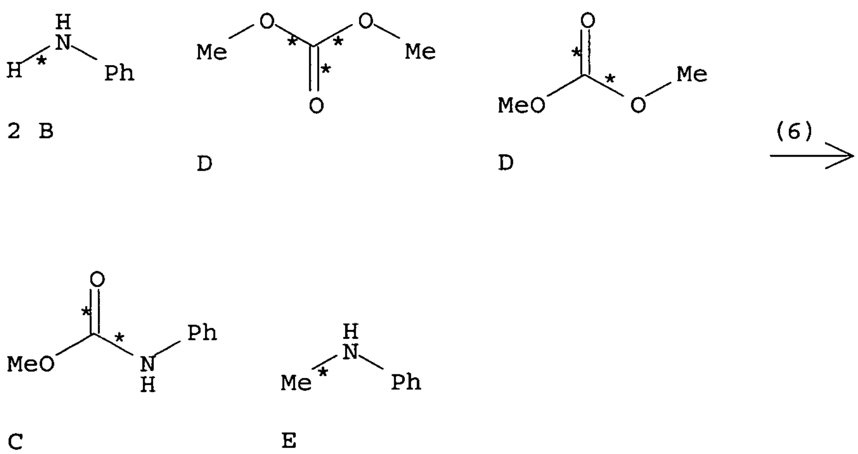
RX(4) RCT B **62-53-3**, D **616-38-6**
PRO E **100-61-8**
CAT 7631-86-9D SiO2

RX(5) OF 6 2 **B** + 2 **D** ===> C + **E**



RX(5) RCT B **62-53-3**, D **616-38-6**
PRO C 2603-10-3, E **100-61-8**
CAT 7631-86-9D SiO2

RX(6) OF 6 2 **B** + 2 **D** ===> C + **E**



RX(6) RCT B **62-53-3**, D **616-38-6**
PRO C 2603-10-3, E **100-61-8**
CAT 7631-86-9D SiO2

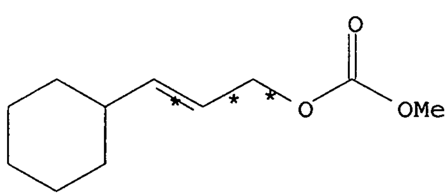
L60 ANSWER 27 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 135:357537 CASREACT
TITLE: Iridium complex-catalyzed allylic amination of allylic esters
AUTHOR(S): Takeuchi, Ryo; Ue, Naoki; Tanabe, Keisuke; Yamashita, Kengo; Shiga, Norihito
CORPORATE SOURCE: Department of Chemistry Graduate School of Integrated Science, Yokohama City University, Kanazawa-ku Yokohama, 236-0027, Japan

SOURCE: Journal of the American Chemical Society (2001), 123(39), 9525-9534
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

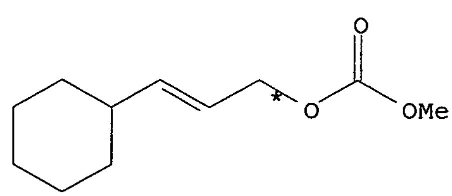
AB Iridium complex-catalyzed allylic amination of allylic carbonates was studied. The solvent strongly affected the catalytic activity. The use of a polar solvent such as EtOH is essential for obtaining the products in high yield. The reaction of (E)-3-substituted-2-propenyl carbonate and 1-substituted-2-propenyl carbonate with pyrrolidine in the presence of a catalytic amount of [Ir(COD)Cl]2 and P(OPh)3 (P/Ir = 2) gave a branched amine with up to 99% selectivity. Both secondary and primary amines could be used for this reaction. When a primary amine was used, selective monoallylation occurred. No diallylation product was obtained. The reaction of 1,1-disubstituted-2-propenyl acetate H2C:CHCR1(OAc)Me (R1 = n-pentyl, Me2C:CHCH2CH2) with amines exclusively gave an α,α-disubstituted allylic amine H2C:CHCR1(NR2)Me (NR2 = piperidino, pyrrolidino, n-butylamino, anilino). This reaction provides an alternative route to the addition of an organometallic reagent to ketimines for the preparation of such amines. The reaction of (Z)-3-substituted-2-propenyl carbonate with amines gave (Z)-linear amines with up to 100% selectivity. In all cases, no (E)-linear amine was obtained. The selectivities described here have not been achieved in similar palladium complex-catalyzed reactions.

REFERENCE COUNT: 120 THERE ARE 120 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

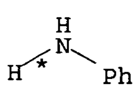
RX(27) OF 45 2 **BT** + 2 **BW** ===> **BX** + BY



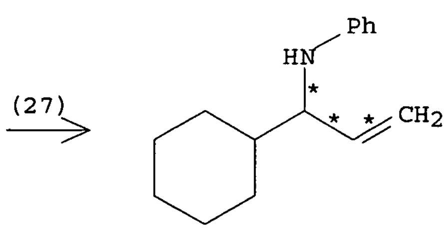
BT



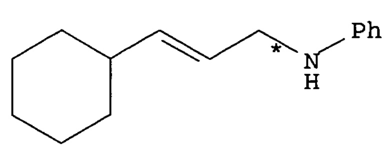
BT



2 BW



BX
YIELD 96%(91)

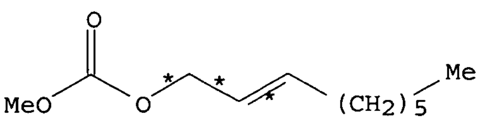


BY
YIELD 96%(9)

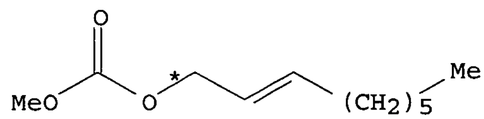
RX(27) RCT BT **373362-32-4**, BW **62-53-3**
PRO BX **373362-42-6**, BY 373362-49-3

CAT 101-02-0 P(OPh)3, 12148-71-9 Iridium, bis[(1,2,5,6-η)-1,5-cyclooctadiene]di-μ-methoxydi-
SOL 64-17-5 EtOH
NTE optimization study, optimized on catalyst and solvent, regioselective, stereoselective

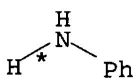
RX(28) OF 45 2 **BE** + 2 **BW** ===> **BZ** + CA



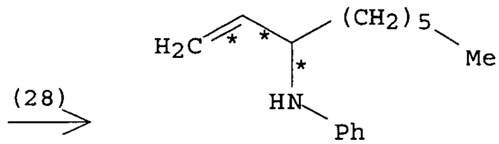
BE



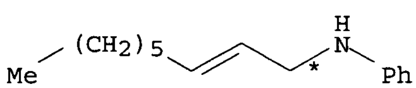
BE



2 BW



BZ
YIELD 95%(93)



CA
YIELD 95%(7)

RX(28) RCT BE **373362-30-2**, BW **62-53-3**
PRO BZ **373362-43-7**, CA 373362-47-1
CAT 101-02-0 P(OPh)3, 12148-71-9 Iridium, bis[(1,2,5,6-η)-1,5-cyclooctadiene]di-μ-methoxydi-
SOL 64-17-5 EtOH
NTE optimization study, optimized on catalyst and solvent, regioselective, stereoselective

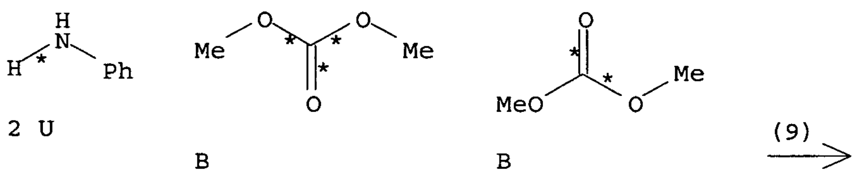
L60 ANSWER 28 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 136:118057 CASREACT
TITLE: 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) and Microwave-Accelerated Green Chemistry in Methylation of Phenols, Indoles, and Benzimidazoles with Dimethyl Carbonate
AUTHOR(S): Shieh, Wen-Chung; Dell, Steven; Repic, Oljan
CORPORATE SOURCE: Chemical and Analytical Development, Novartis Institute for Biomedical Research, East Hanover, NJ, 07936, USA
SOURCE: Organic Letters (2001), 3(26), 4279-4281
CODEN: ORLEF7; ISSN: 1523-7060

PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) is a novel and active catalyst in promoting the methylation reaction of phenols, indoles, and benzimidazoles with di-Me carbonate under mild conditions. Addnl. rate enhancement is accomplished by applying microwave irradiation By incorporating tetrabutylammonium iodide, the same microwave reactions can be further accelerated. By combining these acceleration strategies, very slow chemical transformations that take up to several days can be performed efficiently in high yield within minutes.

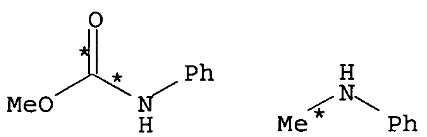
REFERENCE COUNT: 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(9) OF 9 2 **U** + 2 **B** ===> V + **W**



2 U

B B



V
YIELD 76%

W
YIELD 24%

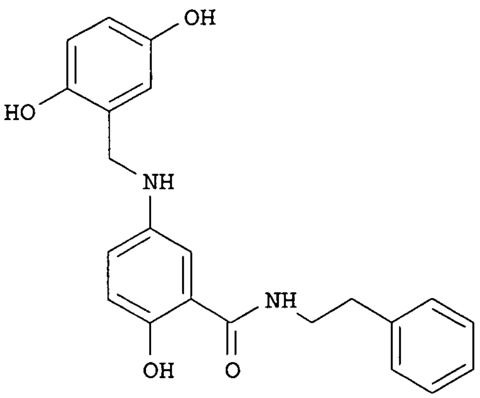
RX(9) RCT U **62-53-3**, B **616-38-6**
PRO V 2603-10-3, W **100-61-8**
CAT 6674-22-2 DBU
NTE thermal, green chem.-reactant

L60 ANSWER 29 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 134:193254 CASREACT
TITLE: Design, Synthesis, and Biological Evaluation of a Series of Lavendustin A Analogues That Inhibit EGFR and Syk Tyrosine Kinases, as Well as Tubulin Polymerization

AUTHOR(S): Mu, Fanrong; Coffing, Stephanie L.; Riese, David J., II; Geahlen, Robert L.; Verdier-Pinard, Pascal; Hamel, Ernest; Johnson, Jill; Cushman, Mark
CORPORATE SOURCE: Department of Medicinal Chemistry and Molecular Pharmacology School of Pharmacy and Pharmacal Sciences, Purdue University, West Lafayette, IN, 47907, USA

SOURCE: Journal of Medicinal Chemistry (2001), 44(3), 441-452
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal
LANGUAGE: English
GI

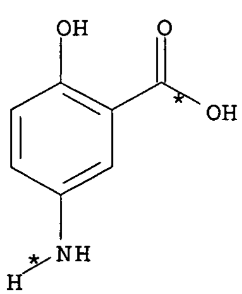


I

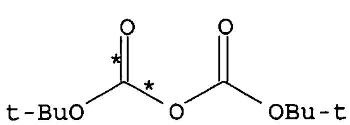
AB A series of N-alkylamide analogs of the lavendustin A pharmacophore, e.g. I, were synthesized and tested for inhibition of the epidermal growth factor receptor (EGFR) protein tyrosine kinase and the nonreceptor protein tyrosine kinase Syk. Although several compds. in the series were effective inhibitors of both kinases, it seemed questionable whether their inhibitory effects on these kinases were responsible for the cytotoxic properties observed in a variety of human cancer cell cultures. Accordingly, a COMPARE anal. of the cytotoxicity profile of the most cytotoxic member of the series was performed, and the results indicated that its cytotoxicity profile was similar to that of antitubulin agents. This mechanism of action was supported by demonstrating that most compds. in the series were moderately effective as inhibitors of tubulin polymerization This suggests that the lavendustin A analogs reported here, as well as some of the previously reported lavendustin A analogs, may be acting as cytotoxic agents by a mechanism involving the inhibition of tubulin polymerization

REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

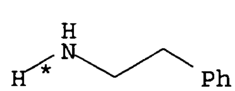
RX(120) OF 156 COMPOSED OF RX(1), RX(2), RX(3), RX(4)
RX(120) **A** + **B** + H + P ===> **Q**



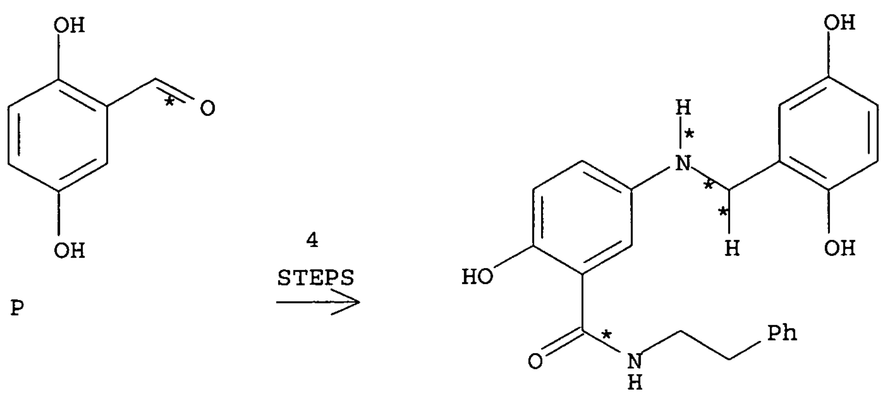
A



B



H



P

Q
YIELD 71%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(2) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT H 64-04-0

STAGE(3)
SOL 7732-18-5 Water

PRO I 327037-08-1

RX(3) RCT I 327037-08-1
RGT N 76-05-1 F3CCO2H
PRO M 327037-09-2
SOL 75-09-2 CH2Cl2

RX(4) RCT M 327037-09-2, P 1194-98-5

STAGE(1)
SOL 71-43-2 Benzene

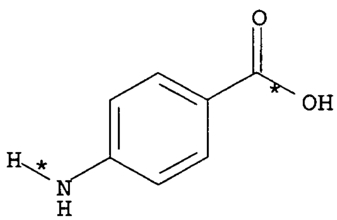
STAGE(2)
RGT R 25895-60-7 NaBH3CN

SOL 67-56-1 MeOH

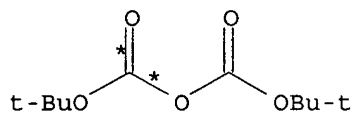
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO Q **327037-10-5**

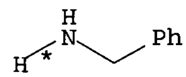
RX(125) OF 156 COMPOSED OF RX(5), RX(6), RX(7), RX(8)
RX(125) **V** + **B** + X + P ===> **AA**



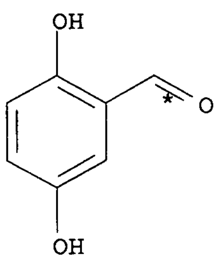
V



B

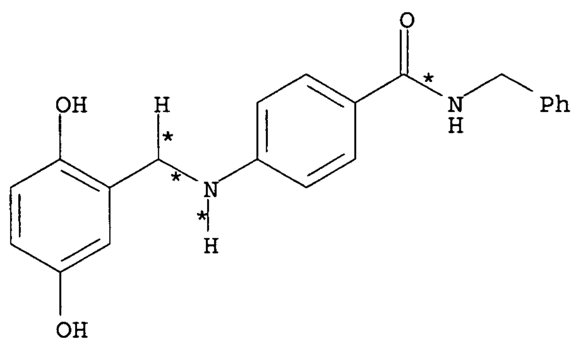


X



P

4 STEPS



AA
YIELD 77%

RX(5) RCT V **150-13-0**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO W 66493-39-8

RX(6) RCT W 66493-39-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8

EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT X 100-46-9

STAGE(3)
SOL 7732-18-5 Water

PRO Y 327037-11-6

RX(7) RCT Y 327037-11-6
RGT N 76-05-1 F3CCO2H
PRO Z 54977-92-3
SOL 75-09-2 CH2Cl2

RX(8) RCT P 1194-98-5, Z 54977-92-3

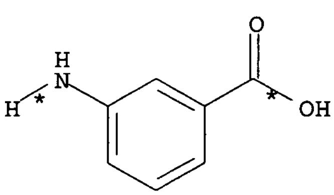
STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

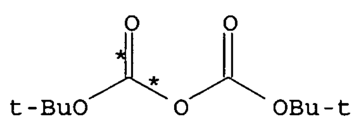
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO AA **327037-12-7**

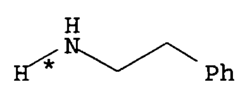
RX(128) OF 156 COMPOSED OF RX(9), RX(10), RX(11), RX(12)
RX(128) **AB** + **B** + H + P ===> **AF**



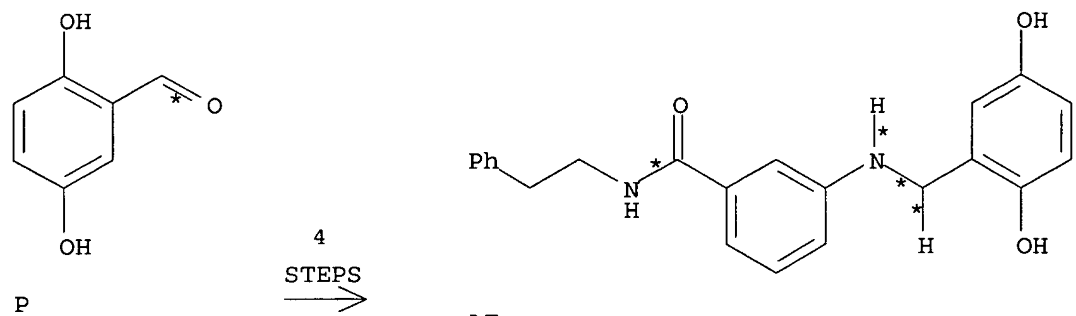
AB



B



H



AF
YIELD 73%

RX(9) RCT AB **99-05-8**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AC 111331-82-9

RX(10) RCT AC 111331-82-9

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT H 64-04-0

STAGE(3)
SOL 7732-18-5 Water

PRO AD 327037-14-9

RX(11) RCT AD 327037-14-9
RGT N 76-05-1 F3CCO2H
PRO AE 81882-72-6
SOL 75-09-2 CH2Cl2

RX(12) RCT P 1194-98-5, AE 81882-72-6

STAGE(1)
SOL 71-43-2 Benzene

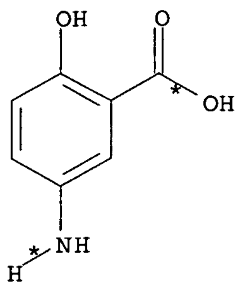
STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl

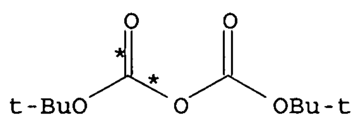
SOL 7732-18-5 Water

PRO AF **185019-03-8**

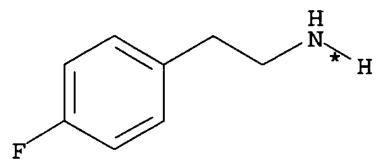
RX(130) OF 156 COMPOSED OF RX(1), RX(13), RX(25), RX(37)
RX(130) **A** + **B** + AG + P ===> **BP**



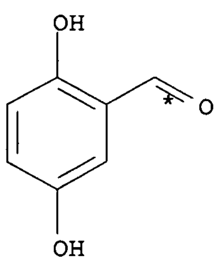
A



B

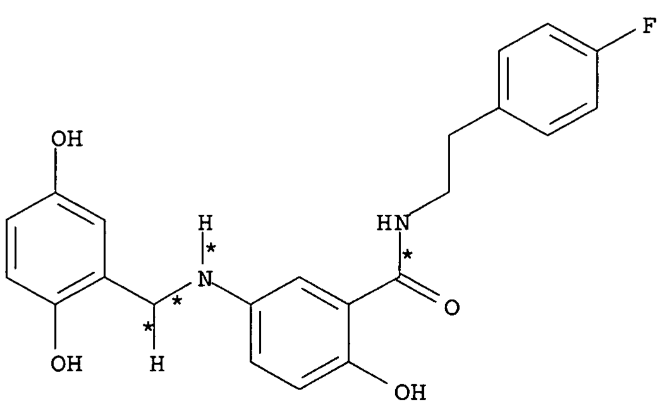


AG



P

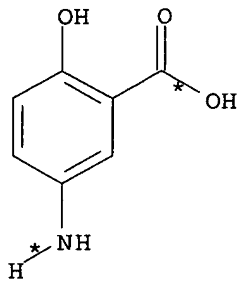
4
STEPS



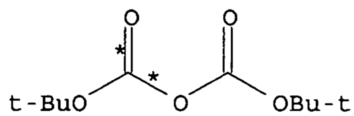
BP
YIELD 42%

RX(1) RCT A **89-57-6**, B **24424-99-5**

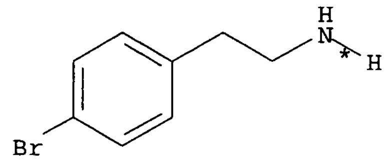
```
          STAGE(1)
             RGT  D 121-44-8 Et3N
             SOL  7732-18-5 Water, 123-91-1 Dioxane

          STAGE(2)
             RGT  E 7647-01-0 HCl
             SOL  7732-18-5 Water

        PRO  C 135321-95-8

RX(13)  RCT  C 135321-95-8

          STAGE(1)
             RGT  D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
                  EDAP
             SOL  68-12-2 DMF

          STAGE(2)
             RCT  AG 1583-88-6

          STAGE(3)
             SOL  7732-18-5 Water

        PRO  AH 327037-15-0

RX(25)  RCT  AH 327037-15-0
        RGT  N 76-05-1 F3CCO2H
        PRO  BD 327037-27-4
        SOL  75-09-2 CH2Cl2

RX(37)  RCT  P 1194-98-5, BD 327037-27-4

          STAGE(1)
             SOL  71-43-2 Benzene

          STAGE(2)
             RGT  R 25895-60-7 NaBH3CN
             SOL  67-56-1 MeOH

          STAGE(3)
             RGT  E 7647-01-0 HCl, S 7647-14-5 NaCl
             SOL  7732-18-5 Water

        PRO  BP 327037-38-7


RX(132) OF 156 COMPOSED OF RX(1), RX(14), RX(26), RX(38)
RX(132)   A  +  B  +  AI  +  P  ===>  BQ
```



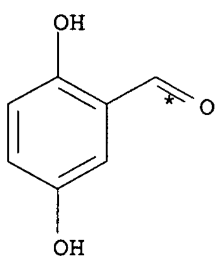
A



B

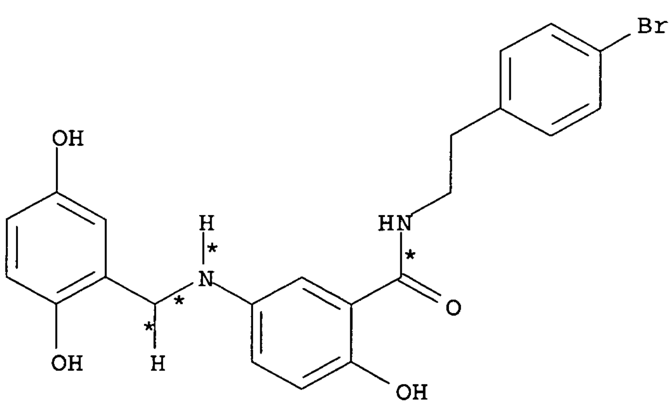


AI



P

4 STEPS
$\longrightarrow$



BQ
YIELD 64%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl

SOL 7732-18-5 Water

PRO C 135321-95-8

RX(14) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AI 73918-56-6

STAGE(3)
SOL 7732-18-5 Water

PRO AJ 327037-16-1

RX(26) RCT AJ 327037-16-1
RGT N 76-05-1 F3CCO2H
PRO BE 327037-28-5
SOL 75-09-2 CH2Cl2

RX(38) RCT P 1194-98-5, BE 327037-28-5

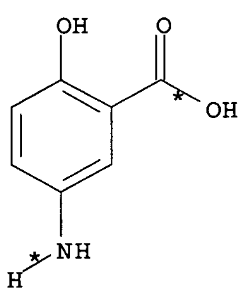
STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

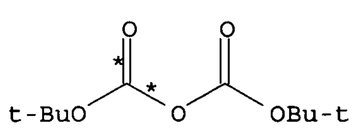
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BQ **327037-39-8**

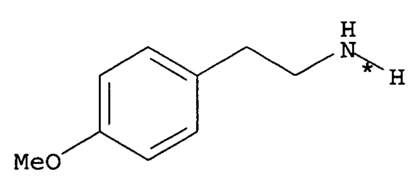
RX(134) OF 156 COMPOSED OF RX(1), RX(15), RX(27), RX(39)
RX(134) **A** + **B** + AK + P ===> **BR**



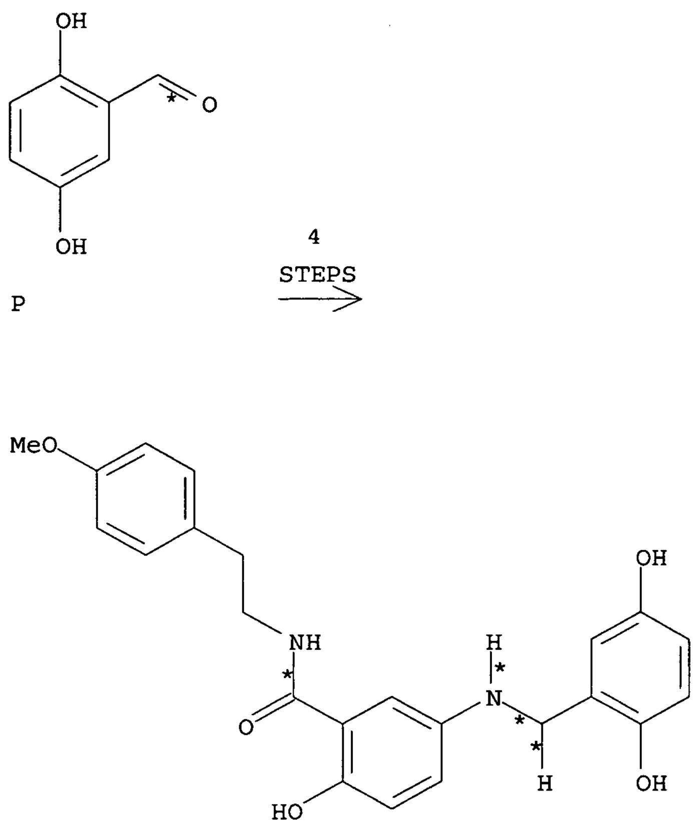
A



B



AK



BR
YIELD 61%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(15) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AK 55-81-2

STAGE(3)
SOL 7732-18-5 Water

PRO AL 327037-17-2

RX(27) RCT AL 327037-17-2
RGT N 76-05-1 F3CCO2H
PRO BF 327037-29-6
SOL 75-09-2 CH2Cl2

RX(39) RCT P 1194-98-5, BF 327037-29-6

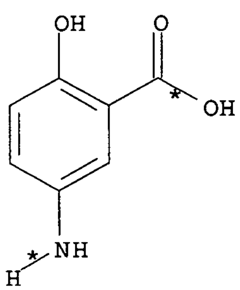
STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

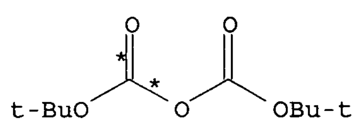
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BR **327037-40-1**

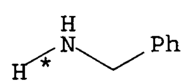
RX(136) OF 156 COMPOSED OF RX(1), RX(16), RX(28), RX(40)
RX(136) **A** + **B** + X + P ===> **BS**



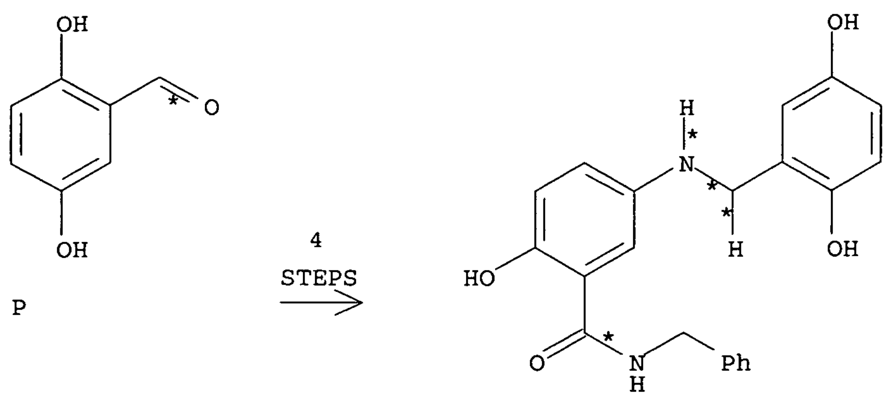
A



B



X



P

BS
YIELD 69%

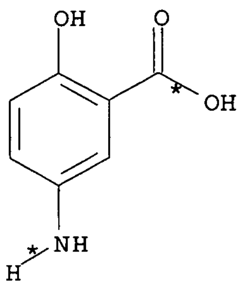
```
RX(1)      RCT  A 89-57-6, B 24424-99-5

             STAGE(1)
                RGT  D 121-44-8 Et3N
                SOL  7732-18-5 Water, 123-91-1 Dioxane

             STAGE(2)
                RGT  E 7647-01-0 HCl
                SOL  7732-18-5 Water

           PRO  C 135321-95-8

RX(16)     RCT  C 135321-95-8

             STAGE(1)
                RGT  D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
                     EDAP
                SOL  68-12-2 DMF

             STAGE(2)
                RCT  X 100-46-9

             STAGE(3)
                SOL  7732-18-5 Water

           PRO  AM 327037-18-3

RX(28)     RCT  AM 327037-18-3
           RGT  N 76-05-1 F3CCO2H
           PRO  BG 154737-59-4
           SOL  75-09-2 CH2Cl2

RX(40)     RCT  P 1194-98-5, BG 154737-59-4

             STAGE(1)
                SOL  71-43-2 Benzene

             STAGE(2)
                RGT  R 25895-60-7 NaBH3CN
```

SOL 67-56-1 MeOH

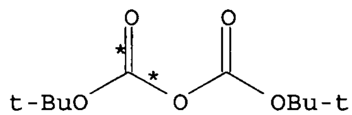
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BS **154736-69-3**

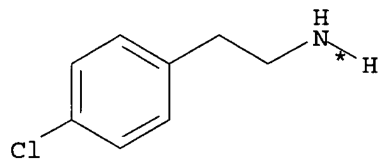
RX(138) OF 156 COMPOSED OF RX(1), RX(17), RX(29), RX(41)
RX(138) **A** + **B** + AN + P ===> **BT**



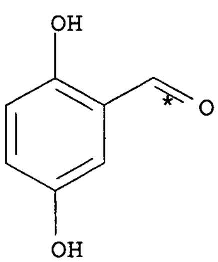
A



B

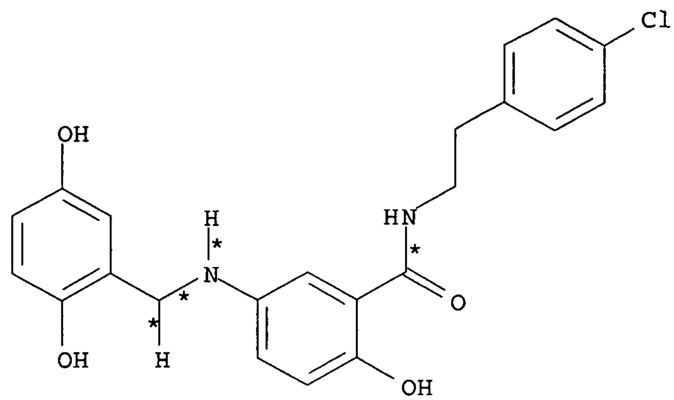


AN



P

$\xrightarrow{\text{4 STEPS}}$



BT
YIELD 69%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(17) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AN 156-41-2

STAGE(3)
SOL 7732-18-5 Water

PRO AO 327037-19-4

RX(29) RCT AO 327037-19-4
RGT N 76-05-1 F3CCO2H
PRO BH 327037-30-9
SOL 75-09-2 CH2Cl2

RX(41) RCT P 1194-98-5, BH 327037-30-9

STAGE(1)
SOL 71-43-2 Benzene

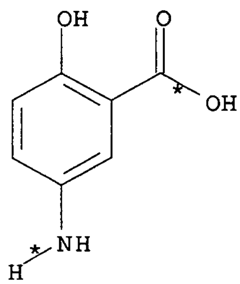
STAGE(2)
RGT R 25895-60-7 NaBH3CN

SOL 67-56-1 MeOH

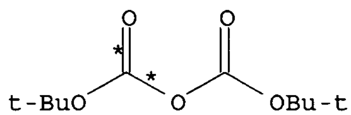
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BT **327037-41-2**

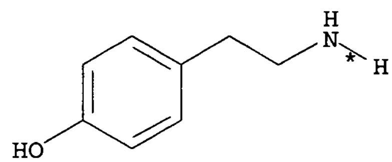
RX(140) OF 156 COMPOSED OF RX(1), RX(18), RX(30), RX(42)
RX(140) **A** + **B** + AP + P ===> **BU**



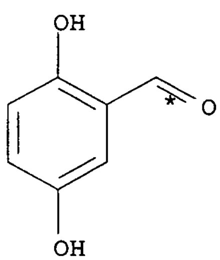
A



B

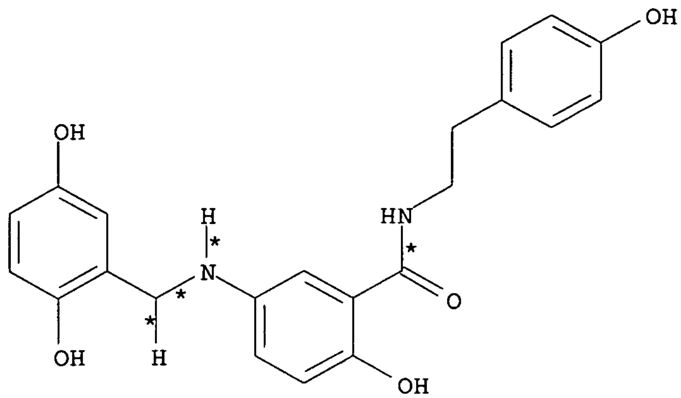


AP



P

4
STEPS



BU
YIELD 55%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(18) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AP 51-67-2

STAGE(3)
SOL 7732-18-5 Water

PRO AQ 327037-20-7

RX(30) RCT AQ 327037-20-7
RGT N 76-05-1 F3CCO2H
PRO BI 327037-31-0
SOL 75-09-2 CH2Cl2

RX(42) RCT P 1194-98-5, BI 327037-31-0

STAGE(1)
SOL 71-43-2 Benzene

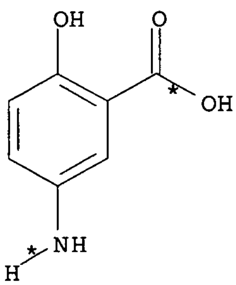
STAGE(2)
RGT R 25895-60-7 NaBH3CN

SOL 67-56-1 MeOH

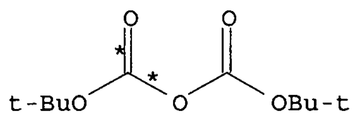
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BU **327037-42-3**

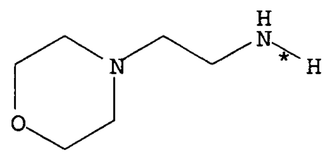
RX(142) OF 156 COMPOSED OF RX(1), RX(19), RX(31), RX(43)
RX(142) **A** + **B** + AR + P ===> **BV**



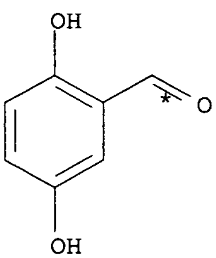
A



B

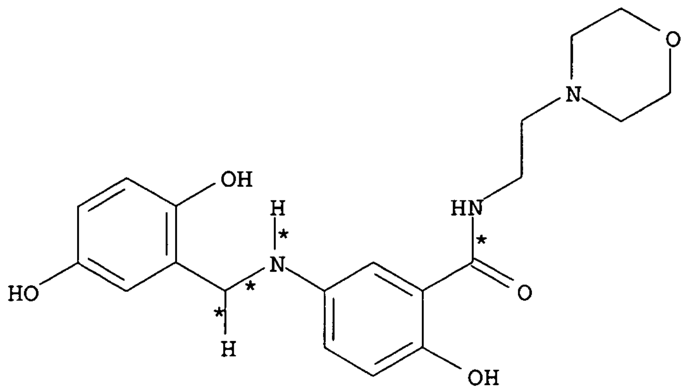


AR



P

4
STEPS
→



BV
YIELD 55%

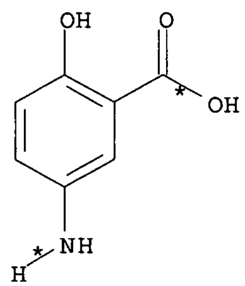
```
RX(1)      RCT  A 89-57-6, B 24424-99-5

             STAGE(1)
                RGT  D 121-44-8 Et3N
                SOL  7732-18-5 Water, 123-91-1 Dioxane

             STAGE(2)
                RGT  E 7647-01-0 HCl
                SOL  7732-18-5 Water

           PRO  C 135321-95-8

RX(19)     RCT  C 135321-95-8

             STAGE(1)
                RGT  D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
                     EDAP
                SOL  68-12-2 DMF

             STAGE(2)
                RCT  AR 2038-03-1

             STAGE(3)
                SOL  7732-18-5 Water

           PRO  AS 327037-21-8

RX(31)     RCT  AS 327037-21-8
           RGT  N 76-05-1 F3CCO2H
           PRO  BJ 327037-32-1
           SOL  75-09-2 CH2Cl2

RX(43)     RCT  P 1194-98-5, BJ 327037-32-1

             STAGE(1)
                SOL  71-43-2 Benzene

             STAGE(2)
                RGT  R 25895-60-7 NaBH3CN
```

SOL 67-56-1 MeOH

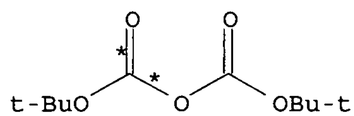
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BV **327037-43-4**

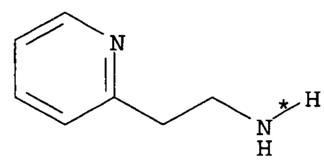
RX(144) OF 156 COMPOSED OF RX(1), RX(20), RX(32), RX(44)
RX(144) **A** + **B** + AT + P ===> **BW**



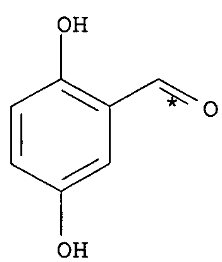
A



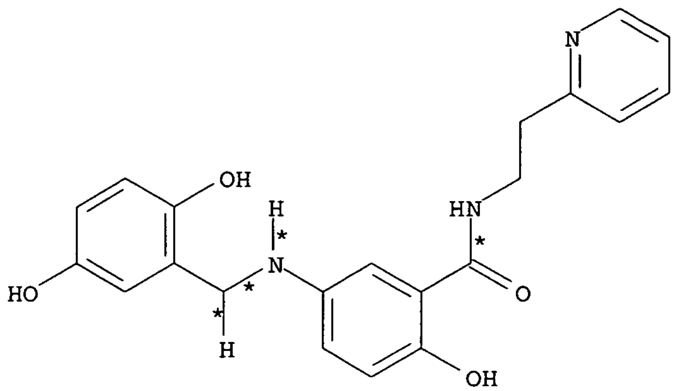
B



AT



P



BW
YIELD 51%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(20) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AT 2706-56-1

STAGE(3)
SOL 7732-18-5 Water

PRO AU 327037-22-9

RX(32) RCT AU 327037-22-9
RGT N 76-05-1 F3CCO2H
PRO BK 327037-33-2
SOL 75-09-2 CH2Cl2

RX(44) RCT P 1194-98-5, BK 327037-33-2

STAGE(1)
SOL 71-43-2 Benzene

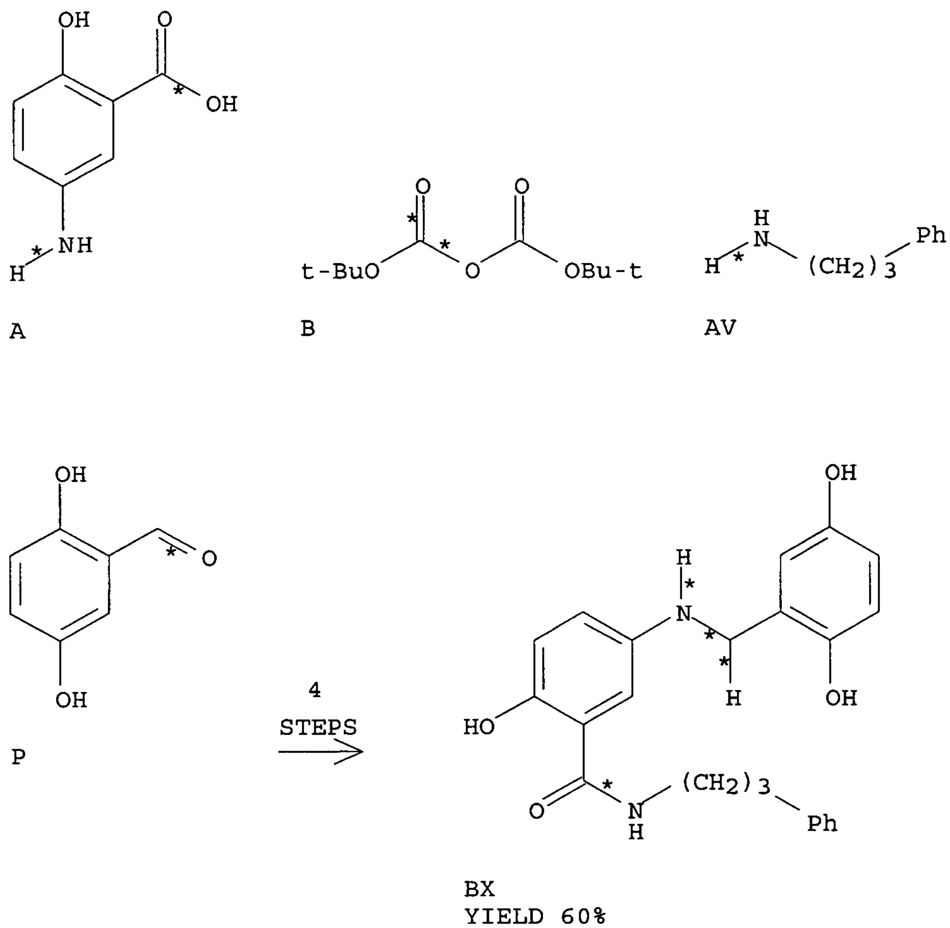
STAGE(2)
RGT R 25895-60-7 NaBH3CN

SOL 67-56-1 MeOH

STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BW **327037-44-5**

RX(146) OF 156 COMPOSED OF RX(1), RX(21), RX(33), RX(45)
RX(146) **A** + **B** + AV + P ===> **BX**



RX(1) RCT A **89-57-6**, B **24424-99-5**

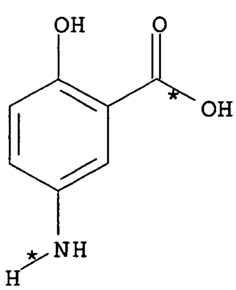
STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

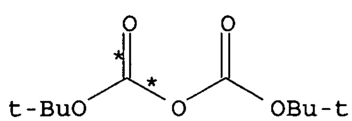
PRO C 135321-95-8

```
RX(21)     RCT  C 135321-95-8

             STAGE(1)
                RGT  D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
                     EDAP
                SOL  68-12-2 DMF

             STAGE(2)
                RCT  AV 2038-57-5

             STAGE(3)
                SOL  7732-18-5 Water

           PRO  AW 327037-23-0

RX(33)     RCT  AW 327037-23-0
           RGT  N 76-05-1 F3CCO2H
           PRO  BL 327037-34-3
           SOL  75-09-2 CH2Cl2

RX(45)     RCT  P 1194-98-5, BL 327037-34-3

             STAGE(1)
                SOL  71-43-2 Benzene

             STAGE(2)
                RGT  R 25895-60-7 NaBH3CN
                SOL  67-56-1 MeOH

             STAGE(3)
                RGT  E 7647-01-0 HCl, S 7647-14-5 NaCl
                SOL  7732-18-5 Water

           PRO  BX 327037-45-6
```

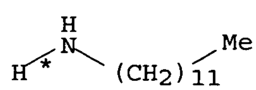
RX(148) OF 156 COMPOSED OF RX(1), RX(22), RX(34), RX(46)
RX(148)   **A** + **B** + AX + P ===> **BY**



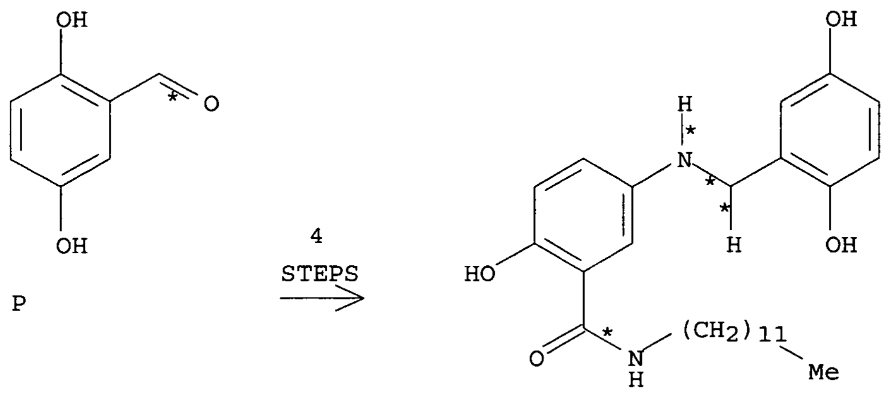
A



B



AX



P

BY
YIELD 56%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(22) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AX 124-22-1

STAGE(3)
SOL 7732-18-5 Water

PRO AY 327037-24-1

RX(34) RCT AY 327037-24-1
RGT N 76-05-1 F3CCO2H
PRO BM 327037-35-4
SOL 75-09-2 CH2Cl2

RX(46) RCT P 1194-98-5, BM 327037-35-4

STAGE(1)
SOL 71-43-2 Benzene

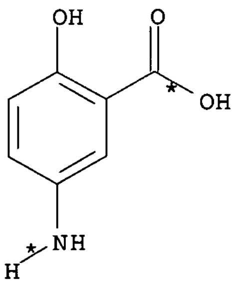
STAGE(2)
RGT R 25895-60-7 NaBH3CN

SOL 67-56-1 MeOH

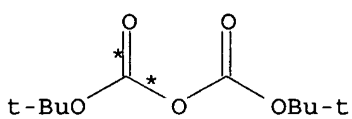
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO BY **327037-46-7**

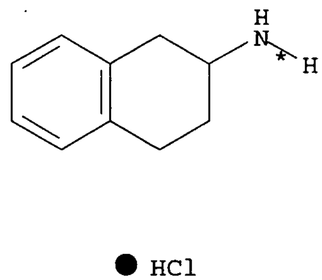
RX(150) OF 156 COMPOSED OF RX(1), RX(23), RX(35), RX(47)
RX(150) **A** + **B** + AZ + P ===> **BZ**



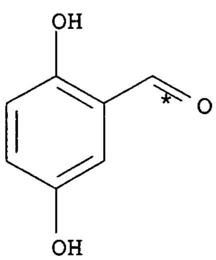
A



B

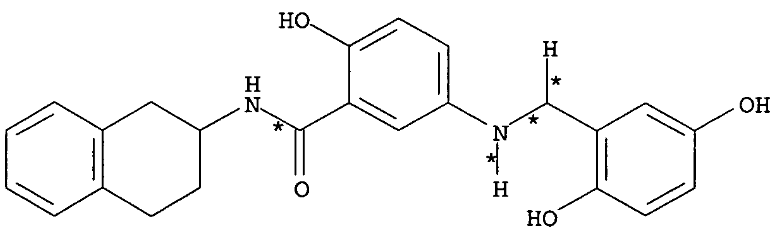


AZ



P

4
STEPS

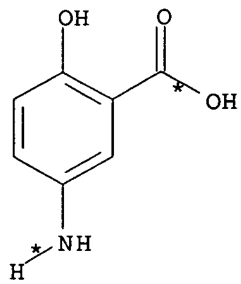


BZ
YIELD 61%

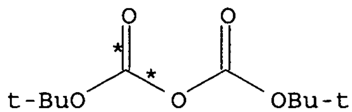
RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)

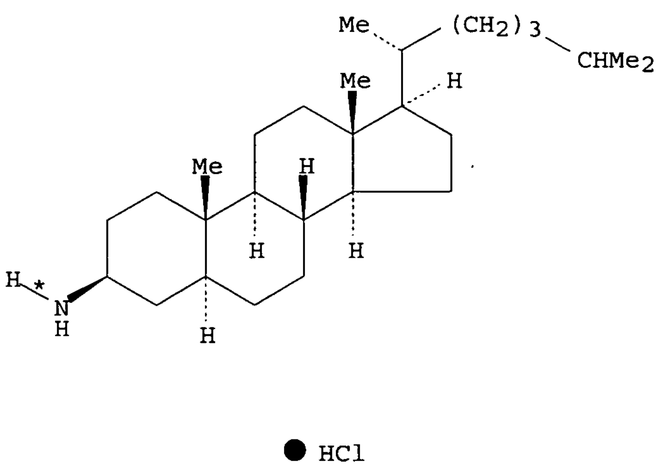
```
            RGT  D 121-44-8 Et3N
            SOL  7732-18-5 Water, 123-91-1 Dioxane

         STAGE(2)
            RGT  E 7647-01-0 HCl
            SOL  7732-18-5 Water

       PRO  C 135321-95-8

RX(23)     RCT  C 135321-95-8

         STAGE(1)
            RGT  D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
                 EDAP
            SOL  68-12-2 DMF

         STAGE(2)
            RCT  AZ 1743-01-7

         STAGE(3)
            SOL  7732-18-5 Water

       PRO  BA 327037-25-2

RX(35)     RCT  BA 327037-25-2
       RGT  N 76-05-1 F3CCO2H
       PRO  BN 327037-36-5
       SOL  75-09-2 CH2Cl2

RX(47)     RCT  P 1194-98-5, BN 327037-36-5

         STAGE(1)
            SOL  71-43-2 Benzene

         STAGE(2)
            RGT  R 25895-60-7 NaBH3CN
            SOL  67-56-1 MeOH

         STAGE(3)
            RGT  E 7647-01-0 HCl, S 7647-14-5 NaCl
            SOL  7732-18-5 Water

       PRO  BZ 327037-47-8


RX(152) OF 156 COMPOSED OF RX(1), RX(24), RX(36), RX(48)
RX(152)    A  +  B  +  BB  +  P  ===>  CA
```



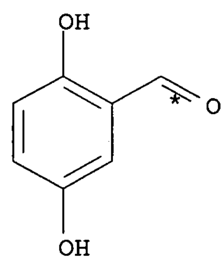
A



B

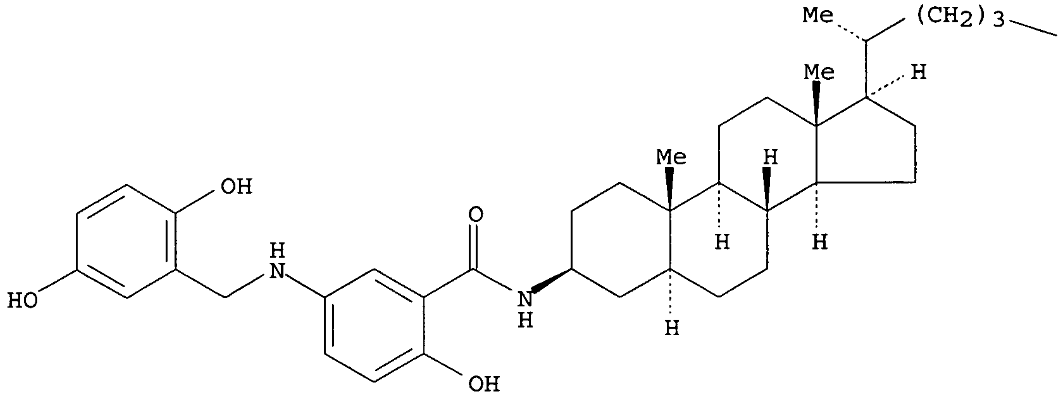


BB



P

PAGE 1-A



PAGE 1-B

CA
YIELD 44%

RX(1) RCT A **89-57-6**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO C 135321-95-8

RX(24) RCT C 135321-95-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT BB 96290-48-1

STAGE(3)
SOL 7732-18-5 Water

PRO BC 327037-26-3

RX(36) RCT BC 327037-26-3
RGT N 76-05-1 F3CCO2H

PRO BO 327037-37-6
SOL 75-09-2 CH2Cl2

RX(48) RCT P 1194-98-5, BO 327037-37-6

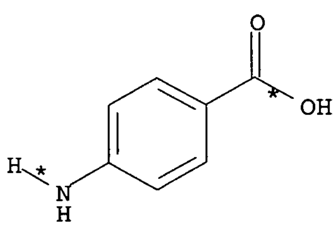
STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

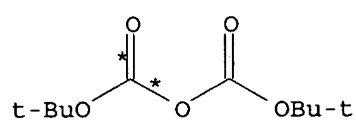
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO CA **327037-48-9**

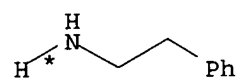
RX(154) OF 156 COMPOSED OF RX(5), RX(49), RX(51), RX(53)
RX(154) **V** + **B** + H + P ===> **CF**



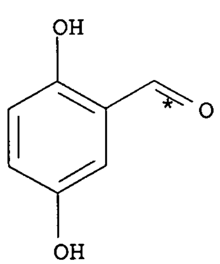
V



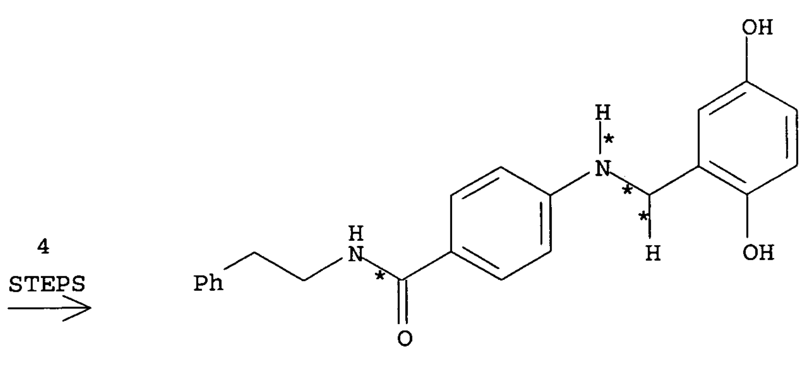
B



H



P



CF
YIELD 59%

RX(5) RCT V **150-13-0**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO W 66493-39-8

RX(49) RCT W 66493-39-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8
EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT H 64-04-0

STAGE(3)
SOL 7732-18-5 Water

PRO CB 327037-49-0

RX(51) RCT CB 327037-49-0
RGT N 76-05-1 F3CCO2H
PRO CD 61251-99-8
SOL 75-09-2 CH2Cl2

RX(53) RCT P 1194-98-5, CD 61251-99-8

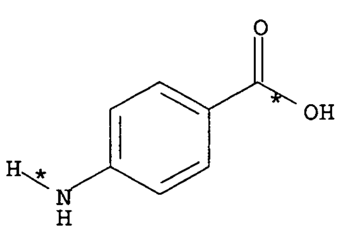
STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

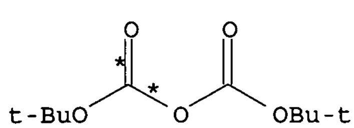
STAGE(3)
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO CF **327037-52-5**

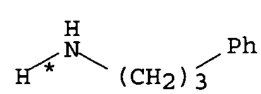
RX(156) OF 156 COMPOSED OF RX(5), RX(50), RX(52), RX(54)
RX(156) **V** + **B** + AV + P ===> **CG**



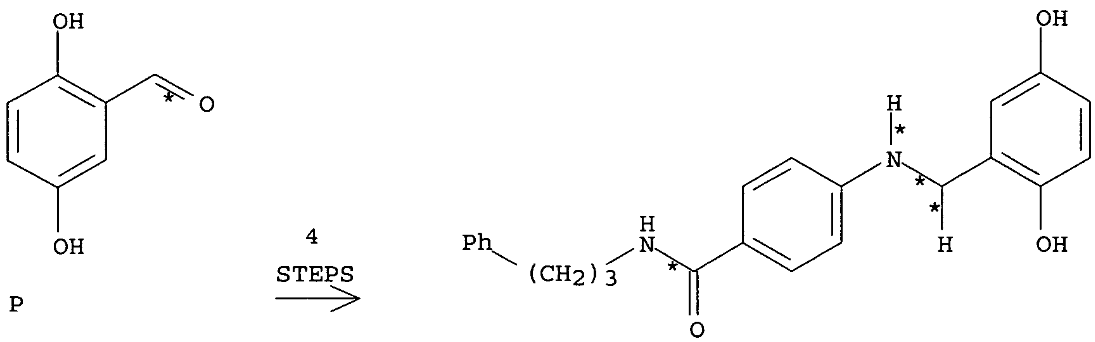
V



B



AV



P

4 STEPS

CG
YIELD 73%

RX(5) RCT V **150-13-0**, B **24424-99-5**

STAGE(1)
RGT D 121-44-8 Et3N
SOL 7732-18-5 Water, 123-91-1 Dioxane

STAGE(2)
RGT E 7647-01-0 HCl
SOL 7732-18-5 Water

PRO W 66493-39-8

RX(50) RCT W 66493-39-8

STAGE(1)
RGT D 121-44-8 Et3N, J 2592-95-2 1-Benzotriazolol, K 25952-53-8 EDAP
SOL 68-12-2 DMF

STAGE(2)
RCT AV 2038-57-5

STAGE(3)
SOL 7732-18-5 Water

PRO CC 327037-50-3

RX(52) RCT CC 327037-50-3
RGT N 76-05-1 F3CCO2H
PRO CE 327037-51-4
SOL 75-09-2 CH2Cl2

RX(54) RCT P 1194-98-5, CE 327037-51-4

STAGE(1)
SOL 71-43-2 Benzene

STAGE(2)
RGT R 25895-60-7 NaBH3CN
SOL 67-56-1 MeOH

STAGE(3)

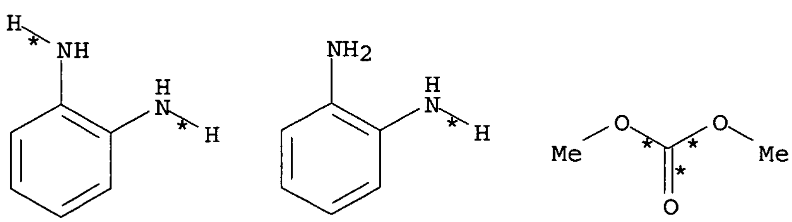
RGT E 7647-01-0 HCl, S 7647-14-5 NaCl
SOL 7732-18-5 Water

PRO CG **327037-53-6**

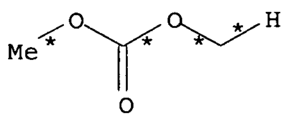
L60 ANSWER 30 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 134:252292 CASREACT
TITLE: Carbonylation of o-Phenylenediamine and o-Aminophenol with Dimethyl Carbonate Using Lead Compounds as Catalysts
AUTHOR(S): Fu, Yue; Baba, Toshihide; Ono, Yoshio
CORPORATE SOURCE: Department of Chemical Engineering, Tokyo Institute of Technology, Ookayama, Meguro-ku, Tokyo, 152-8552, Japan
SOURCE: Journal of Catalysis (2001), 197(1), 91-97
CODEN: JCTLA5; ISSN: 0021-9517
PUBLISHER: Academic Press
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Lead compds. are active catalysts for carbonylation and carbonylation/methylation of o-phenylenediamine and o-aminophenol with di-Me carbonate (DMC). 2-Benzimidazolone was obtained in 84% yield by the reaction of o-phenylenediamine with DMC for 1 h at 443 K in the presence of Pb(NO3)2. In the presence of Pb(OAc)2, the reaction quant. gave 1,3-dimethyl-2-benzimidazolone, which was formed by methylation of the primary product, 2-benzimidazolone, at 473 K. The effects of reaction variables on the yields of 2-benzimidazolone and 1,3-dimethyl-2-benzimidazolone were examined The reaction of o-aminophenol with DMC selectively gave a carbonylation product, 2-benzoxazolone, or a carbonylation/methylation product, 3-methyl-2-benzoxazolone, depending on the reaction conditions in the presence of Pb(OAc)2. (c) 2001 Academic Press.
REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 7 3 **A** + 3 **B** ===> C + D + **E**

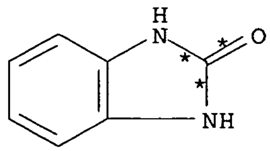


2 A A 2 B

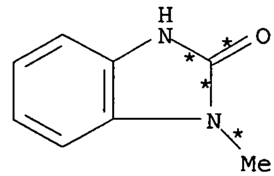


B

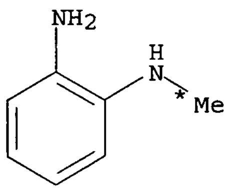
(1) →



C
YIELD 77%



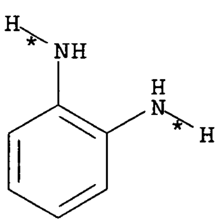
D
YIELD 10%



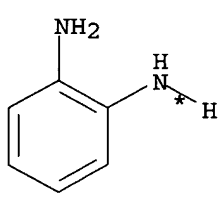
E
YIELD 13%

RX(1) RCT A **95-54-5**, B **616-38-6**
PRO C 615-16-7, D 1849-01-0, E **4760-34-3**
CAT 10099-74-8 Pb(NO3)2
NTE no solvent, alternative catalysts gave lower yields

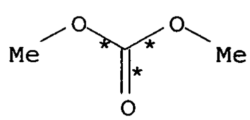
RX(5) OF 7 2 **A** + 2 **B** ===> C + **E**



A

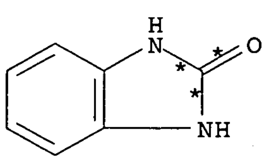


A

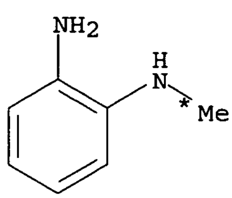


2 B

(5) →



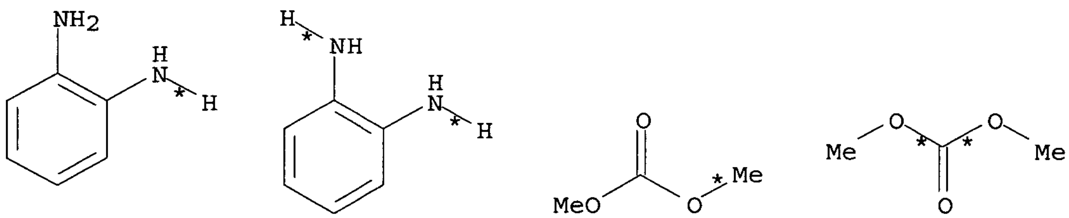
C
YIELD 5%



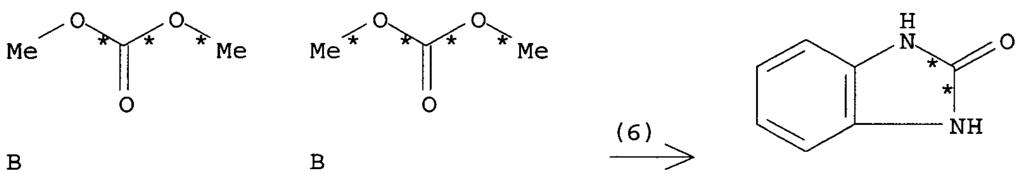
E
YIELD 75%

RX(5) RCT A **95-54-5**, B **616-38-6**
PRO C 615-16-7, E **4760-34-3**
NTE no solvent, use of catalysts gave lower yields

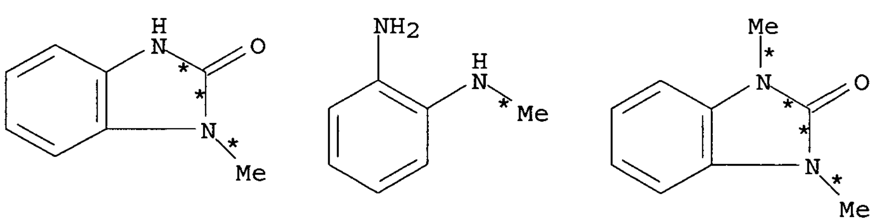
RX(6) OF 7 4 **A** + 4 **B** ===> C + D + **E**
+ L



A 3 A B B



B B C
YIELD 23%



D
YIELD 39%

E
YIELD 9%

L
YIELD 29%

RX(6) RCT A **95-54-5**, B **616-38-6**
PRO C 615-16-7, D 1849-01-0, E **4760-34-3**, L 3097-21-0
CAT 10099-74-8 Pb(NO3)2
NTE no solvent, alternative catalysts gave lower yields

L60 ANSWER 31 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 132:207657 CASREACT
TITLE: Preparation of linkers for solid phase synthesis
INVENTOR(S): Nagai, Katsunori; Miwa, Tetsuo
PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan

SOURCE: Eur. Pat. Appl., 42 pp.
CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

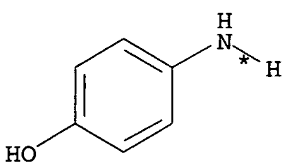
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 985662	A2	20000315	EP 1999-117080	19990831
EP 985662	A3	20030312		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
US 6388022	B1	20020514	US 1999-385953	19990830
CA 2281490	AA	20000310	CA 1999-2281490	19990909
CN 1247863	A	20000322	CN 1999-118556	19990909
KR 2000023020	A	20000425	KR 1999-38336	19990909
JP 2000143553	A2	20000523	JP 1999-255207	19990909
PRIORITY APPLN. INFO.:			JP 1998-257264	19980910

OTHER SOURCE(S): MARPAT 132:207657

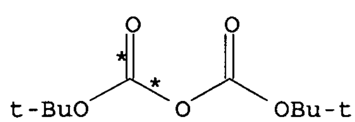
AB RYZW(SO2X)m [R = a carrier; X = leaving group; Y = bond or spacer; Z = bivalent electron-attracting group and W = (un)substituted di- or trivalent aromatic ring; Z = bivalent non-electron-attracting group and W = electron-attracting group-substituted aromatic ring; m = 1 or 2] were prepared and their use in solid phase synthesis demonstrated. Thus, 4-RC6H4SO2C6H4(SO2Cl)-3 (preparation given) was esterified by 4-(HO)C6H4NHCO2CMe3 and the deprotected product reductively alkylated by PhCHO to give 3-(4-RC6H4SO2)C6H4SO3C6H4(NHPh)-4 which was N-acylated by 3,4,5-trimethoxybenzoyl chloride and the product treated with Pd(OAc)2/Ph2P(CH2)3PPh2/NEt3/HCO2H to give PhCH2NPHCOC6H2(OMe)3-3,4,5.

RX(21) OF 30 COMPOSED OF RX(7), RX(1), RX(3), RX(4)

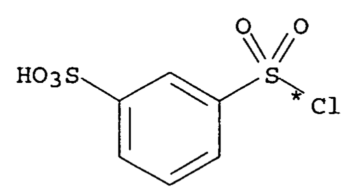
RX(21) **Z** + **AA** + B + L ===> **M**



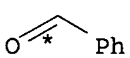
Z



AA

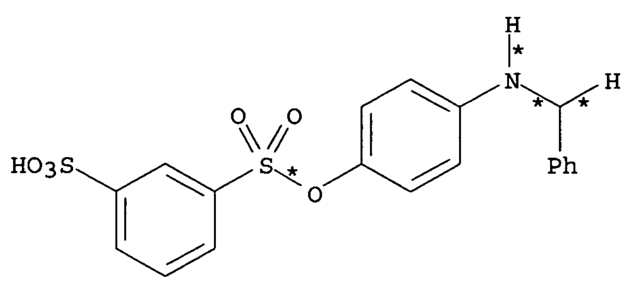


B



L

4 STEPS



M

```
RX(7)     RCT  Z 123-30-8, AA 24424-99-5
          PRO  A 54840-15-2
          SOL  109-99-9 THF

RX(1)     RCT  A 54840-15-2, B 148344-35-8D
          RGT  D 121-44-8 Et3N
          PRO  C 260396-17-6D
          SOL  109-99-9 THF
          NTE  RESIN SUPPORTED REACTION

RX(3)     RCT  C 260396-17-6D
          RGT  J 76-05-1 F3CCO2H
          PRO  I 260396-18-7D
          SOL  75-09-2 CH2Cl2
          NTE  RESIN SUPPORTED REACTION

RX(4)     RCT  I 260396-18-7D, L 100-52-7

            STAGE(1)
               SOL  75-09-2 CH2Cl2

            STAGE(2)
               RGT  N 64-19-7 AcOH, O 56553-60-7 Na.(AcO)3BH

            STAGE(3)
               SOL  68-12-2 DMF, 67-56-1 MeOH, 109-99-9 THF, 60-29-7 Et2O

          PRO  M 260396-19-8D
          NTE  RESIN SUPPORTED REACTION
```

L60 ANSWER 32 OF 44 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 134:4719 CASREACT

TITLE: Selective methylation of phenol, aniline and catechol with dimethyl carbonate over calcined Mg-Al hydrotalcites

AUTHOR(S): Jyothi, T. M.; Raja, T.; Talawar, M. B.; Sreekumar, K.; Sugunan, S.; Rao, B. S.

CORPORATE SOURCE: National Chemical Laboratory, Pune, 411 021, India

SOURCE: Synthetic Communications (2000), 30(21), 3929-3934
CODEN: SYNCAV; ISSN: 0039-7911

PUBLISHER: Marcel Dekker, Inc.

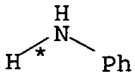
DOCUMENT TYPE: Journal

LANGUAGE: English

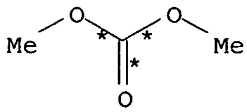
AB Calcined Mg-Al hydrotalcites (Mg/Al = 3) can be used as an efficient catalyst in the selective O-methylation of phenol and catechol and N-monomethylation of aniline employing (MeO)2CO as methylating agent in vapor phase at 275°.

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(2) OF 3 2 **E** + 3 **B** ===> **F** + G

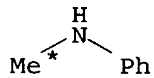


2 E

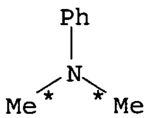


3 B

(2) →



F
YIELD 85%



G
YIELD 10%

RX(2) RCT E **62-53-3**, B **616-38-6**
PRO F **100-61-8**, G 121-69-7
CAT 135752-28-2 Aluminum magnesium carbonate hydroxide
NTE solid-support, silica

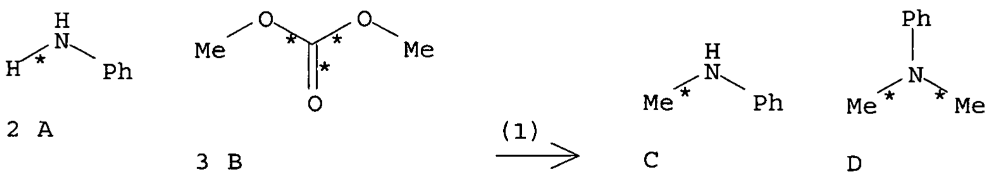
L60 ANSWER 33 OF 44 CASREACT

ACCESSION NUMBER: 134:4715 CASREACT
TITLE: Selective N-methylation of aniline with dimethyl carbonate over Zn1-xCoxFe2O4 (x = 0, 0.2, 0.5, 0.8 and 1.0) type systems
AUTHOR(S): Sreekumar, K.; Jyothi, T. M.; Mathew, T.; Talawar, M. B.; Sugunan, S.; Rao, B. S.
CORPORATE SOURCE: Department of Applied Chemistry, Cochin University of Science and Technology, Cochin, 682 022, India
SOURCE: Journal of Molecular Catalysis A: Chemical (2000), 159(2), 327-334
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The alkylation of aniline with di-Me carbonate (DMC) is reported over Zn1-xCoxFe2O4 (x = 0, 0.2, 0.5, 0.8 and 1.0) type systems prepared via. the copptn. route. The influence of surface acid-base properties, cation distribution in the spinel lattice and various reaction parameters are discussed. It was observed that systems possessing low x values are highly selective and active for N-alkylation leading to N-methylaniline as the major product. Since the authors have already reported the alkylation reaction using methanol as the alkylating agent over the same ferrospinel systems, in some cases the data are compared to highlight the merits and demerits of the choice of alkylating agent. DMC acts as a better alkylating agent at comparatively low temperature, where methanol shows only mild activity. However, on the selectivity basis DMC as an alkylating agent could not compete with methanol, since the former produced appreciable amount of N,N-dimethylaniline even at low temperature where methanol gave nearly 99% NMA selectivity. Cation distribution in the spinel lattice influences its acido-basic properties, and hence, these factors have been considered as helpful to evaluate the activity and stability of

the systems.
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 1 2 **A** + 3 **B** ===> **C** + D

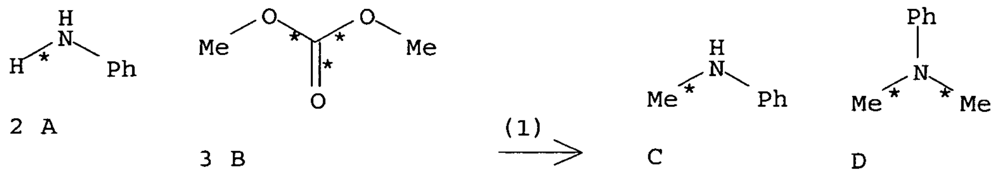


RX(1) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 220942-91-6 Iron zinc oxide (Fe3ZnO4)
NTE chemoselective, optimization study, catalyst compn. affects amt. of mono and di-N-alkylated products formed

L60 ANSWER 34 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 133:207620 CASREACT
TITLE: A comparative study on aniline alkylation activity using methanol and dimethyl carbonate as the alkylating agents over Zn-Co-Fe ternary spinel systems
AUTHOR(S): Sreekumar, K.; Mathew, T.; Mirajkar, S. P.; Sugunan, S.; Rao, B. S.
CORPORATE SOURCE: Department of Applied Chemistry, Cochin University of Science & Technology, Cochin, India
SOURCE: Applied Catalysis, A: General (2000), 201(1), L1-L8
CODEN: ACAGE4; ISSN: 0926-860X
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English

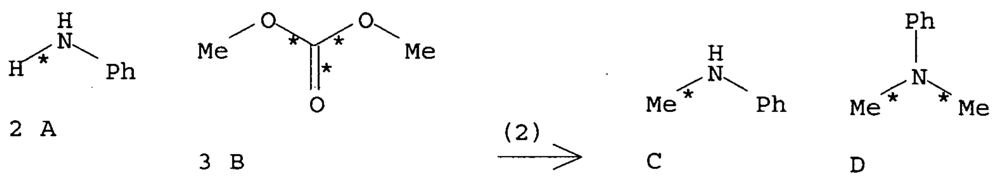
AB The catalyst compns. of the Zn1-xCoxFe2O4 (x= 0, 0.2, 0.5, 0.8 and 1.0) spinel series possessing 'x' values, x≤0.5, are unique for selective N-monomethylation of aniline using methanol as the alkylating agent. Catalysts thus prepared were Iron zinc oxide (Fe2ZnO4), cobalt iron zinc oxide (Co0.2Fe2Zn0.8O4), cobalt iron zinc oxide (Co0.5Fe2Zn0.5O4), cobalt iron zinc oxide (Co0.8Fe2Zn0.2O4), and cobalt iron oxide (CoFe2O4). Since di-Me carbonate (DMC) is another potential non-toxic alkylating agent, alkylation of aniline was investigated over various Zn-Co ferrites using DMC as the alkylating agent. The merits and demerits of the two alkylating agents are compared. Catalytic activity followed a similar trend with respect to the composition of the ferrospinel systems. DMC is active at comparatively low temperature, where methanol shows only mild activity. However, on the selectivity basis, DMC as an alkylating agent could not compete with methanol, since the former gave appreciable amts. of N,N-dimethylaniline (NNDMA) even at low temperature where methanol gave nearly 99% N-methylaniline (NMA) selectivity. As in the case of methanol, DMC also did not give any C-alkylated products.
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 9 2 **A** + 3 **B** ===> **C** + D



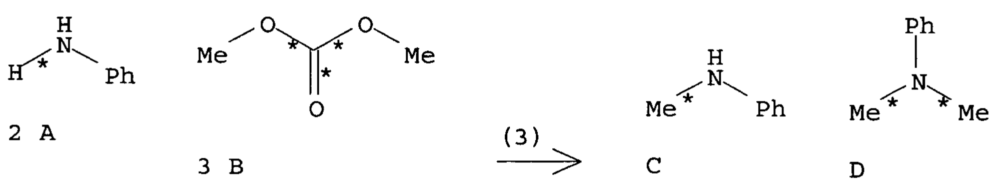
RX(1) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 12063-19-3 Iron zinc oxide (Fe_2ZnO_4)
NTE vapor-phase down-flow silica reactor; (75.3:10.9 mono:di)

RX(2) OF 9 2 **A** + 3 **B** ===> **C** + D



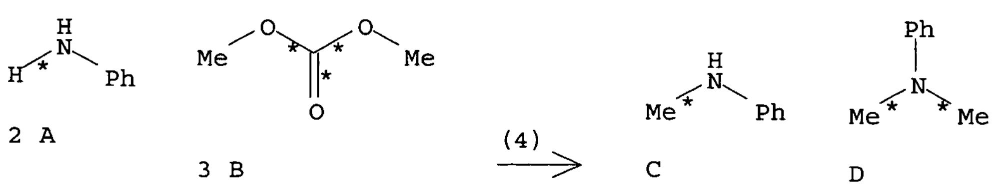
RX(2) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 118033-24-2 Cobalt iron zinc oxide ($Co_{0.2}Fe_2Zn_{0.8}O_4$)
NTE vapor-phase down-flow silica reactor; (73.8:11.9 mono:di)

RX(3) OF 9 2 **A** + 3 **B** ===> **C** + D



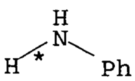
RX(3) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 111568-68-4 Cobalt iron zinc oxide ($Co_{0.8}Fe_2Zn_{0.2}O_4$)
NTE vapor-phase down-flow silica reactor; (62.2:20.1 mono:di)

RX(4) OF 9 2 **A** + 3 **B** ===> **C** + D

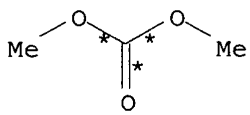


RX(4) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 12052-28-7 Cobalt iron oxide (CoFe2O4)
NTE vapor-phase down-flow silica reactor; (61.2:21.1 mono:di)

RX(5) OF 9 2 **A** + 3 **B** ===> **C** + D

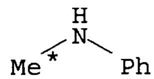


2 A

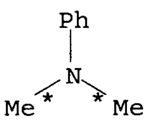


3 B

(5) →



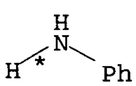
C
YIELD 71%



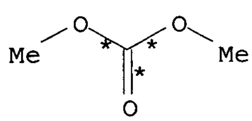
D
YIELD 7%

RX(5) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 12063-19-3 Iron zinc oxide (Fe2ZnO4)
NTE vapor-phase down-flow silica reactor; yields vary with temp.

RX(6) OF 9 2 **A** + 3 **B** ===> **C** + D

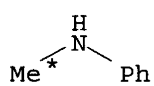


2 A

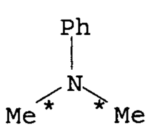


3 B

(6) →



C
YIELD 67%

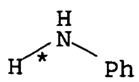
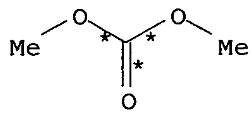
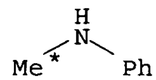


D
YIELD 5%

RX(6) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 118033-24-2 Cobalt iron zinc oxide (Co0.2Fe2Zn0.8O4)

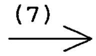
NTE vapor-phase down-flow silica reactor; yields vary with temp.

RX(7) OF 9 2 **A** + 3 **B** ===> **C** + D

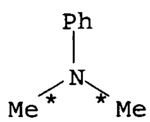




2 A

3 B

(7)


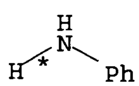
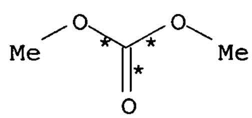
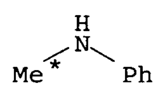
C
YIELD 44%



D
YIELD 9%

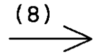
RX(7) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 111568-47-9 Cobalt iron zinc oxide (Co0.5Fe2Zn0.5O4)
NTE vapor-phase down-flow silica reactor; yields vary with temp.

RX(8) OF 9 2 **A** + 3 **B** ===> **C** + D

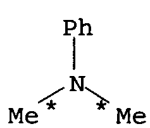




2 A

3 B

(8)


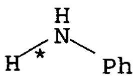
C
YIELD 24%



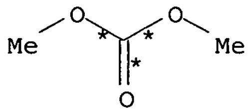
D
YIELD 4%

RX(8) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 111568-68-4 Cobalt iron zinc oxide (Co0.8Fe2Zn0.2O4)
NTE vapor-phase down-flow silica reactor; yields vary with temp.

RX(9) OF 9 2 **A** + 3 **B** ===> **C** + D

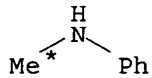


2 A

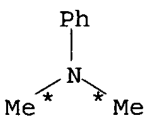


3 B

(9) ——>



C
YIELD 8%



D
YIELD 2%

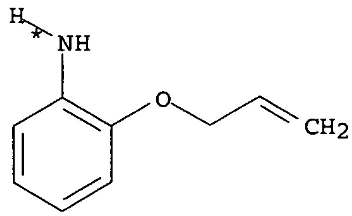
RX(9) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**, D 121-69-7
CAT 12052-28-7 Cobalt iron oxide (CoFe2O4)
NTE vapor-phase down-flow silica reactor; yields vary with temp.

L60 ANSWER 35 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 129:216469 CASREACT
TITLE: Ligand effects in the metal catalyzed reactions of N-aryldiazoamides: ylide formation vs. insertion reactions
AUTHOR(S): Moody, Christopher J.; Miah, Soyfur; Slawin, Alexandra M. Z.; Mansfield, Darren J.; Richards, Ian C.
CORPORATE SOURCE: Dep. Chem., Loughborough Univ., Leicestershire, LE11 3TU, UK
SOURCE: Tetrahedron (1998), 54(33), 9689-9700
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English

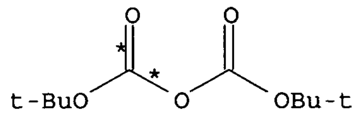
AB Diazoamides 2-CH2:CHCH2XC6H4N(CH2Ph)COC(:N2)CO2Et (I, X = O, S) were prepared from 2-(O-allyl)- and 2-(S-allyl) anilines, resp. Copper(II) catalyzed decomposition of I resulted in formation of benzo-fused heterocycles by [2,3]-rearrangement of the intermediate ylide. Rhodium(II) perfluorobutyramide catalyzed reaction, however, gave mainly indoles; rhodium(II) acetate catalyzed reaction resulted in a mixture of products, with β-lactams formed by intramol. C-H insertion predominating. Catalyzed decomposition of a N-pyridyl diazoamide gave a stable pyridinium ylide.

REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

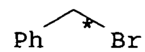
RX(34) OF 65 COMPOSED OF RX(1), RX(3), RX(5)
RX(34) **A** + **B** + G ===> **M**



A

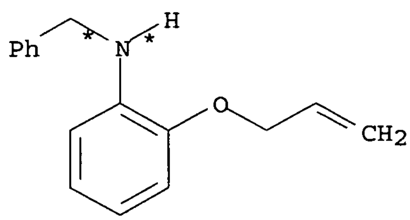


B



G

3 STEPS →



M
YIELD 94%

RX(1) RCT A **27096-64-6**, B **24424-99-5**
PRO C 212475-56-4
SOL 109-99-9 THF

RX(3) RCT C 212475-56-4

STAGE(1)
RGT I 7646-69-7 NaH
SOL 68-12-2 DMF

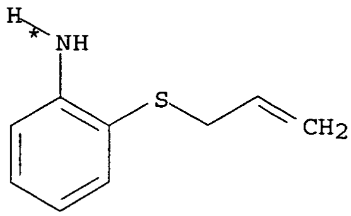
STAGE(2)
RCT G 100-39-0

STAGE(3)
RGT J 7732-18-5 Water

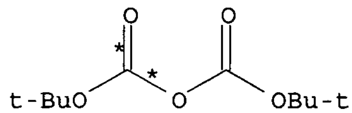
PRO H 212475-60-0

RX(5) RCT H 212475-60-0
RGT N 76-05-1 F3CCO2H
PRO M **212475-64-4**
SOL 75-09-2 CH2Cl2

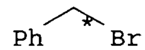
RX(35) OF 65 COMPOSED OF RX(2), RX(4), RX(6)
RX(35) **E** + **B** + G ===> **P**



E

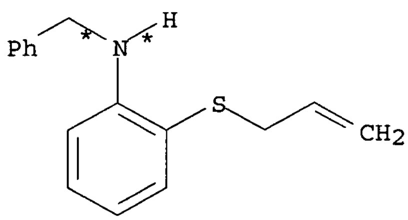


B



G

3
STEPS
→



P
YIELD 99%

```
RX(2)     RCT  E 77053-20-4, B 24424-99-5
          PRO  F 212475-58-6
          SOL  109-99-9 THF

RX(4)     RCT  F 212475-58-6

            STAGE(1)
               RGT  I 7646-69-7 NaH
               SOL  68-12-2 DMF

            STAGE(2)
               RCT  G 100-39-0

            STAGE(3)
               RGT  J 7732-18-5 Water

          PRO  L 212475-62-2

RX(6)     RCT  L 212475-62-2
          RGT  N 76-05-1 F3CCO2H
          PRO  P 212475-66-6
          SOL  75-09-2 CH2Cl2
```

L60 ANSWER 36 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 123:228227 CASREACT

TITLE: Preparation of 1-acylmethyl-2-oxo-3-phenylureido-5-heterocyclyl-1,4-benzodiazepines useful as CCK-B and/or gastrin receptor antagonists.

INVENTOR(S): Semple, Graeme; Ryder, Hamish; Szelke, Michael; Satoh, Masato; Ohta, Mitsuaki; Miyata, Keiji; Nishida, Akito; Ishii, Masato

PATENT ASSIGNEE(S): Yamanouchi Pharmaceutical Co. Ltd., Japan; Ferring Research Ltd.

SOURCE: PCT Int. Appl., 77 pp.
CODEN: PIXXD2

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9506040	A1	19950302	WO 1994-GB1859	19940825
W: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LT, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, US, UZ, VN				
RW: KE, MW, SD, AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
GB 2282595	A1	19950412	GB 1993-17693	19930825
CA 2169089	AA	19950302	CA 1994-2169089	19940825
AU 9474661	A1	19950321	AU 1994-74661	19940825
AU 687433	B2	19980226		
ZA 9406474	A	19960325	ZA 1994-6474	19940825
EP 715624	A1	19960612	EP 1994-924368	19940825
EP 715624	B1	19980408		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE				
CN 1129442	A	19960821	CN 1994-193134	19940825
HU 73978	A2	19961028	HU 1996-205	19940825
JP 09504005	T2	19970422	JP 1994-507439	19940825
AT 164840	E	19980415	AT 1994-924368	19940825
ES 2117797	T3	19980816	ES 1994-924368	19940825
FI 9600836	A	19960422	FI 1996-836	19960223
NO 9600747	A	19960425	NO 1996-747	19960223
√US 5728829	A	19980317	US 1996-591567	19960502
PRIORITY APPLN. INFO.:			GB 1993-17693	19930825
			WO 1994-GB1859	19940825

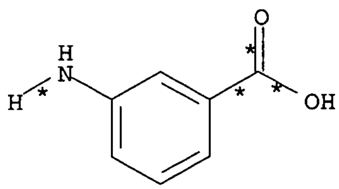
OTHER SOURCE(S): MARPAT 123:228227

GI

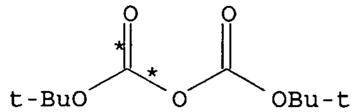
* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Title compds. [I; R4 = alkyl, cycloalkyl, aryl; R10 = halo, OH, Me, OMe, NR11R12, NO2, NHCHO, CO2H, cyano; R11, R12 = H, alkyl; NR11R12 = Q1; a = 1-6; R2 = aromatic 5- or 6-membered (substituted) heterocyclyl containing ≥2 heteroatoms of which ≥1 is N], were prepared Thus, title compound (II), prepared from 2-aminophenyl 2-thiazolyl ketone via 3-amino-1-tert-butylcarbonylmethyl-2,3-dihydro-5-(2-thiazolyl)-1H-1,4-benzodiazepin-2-one, at 0.1 μmol/kg in rats inhibited pentagastrin-stimulated gastric acid secretion by 55.2%. Tablets were prepared containing II.

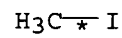
RX(284) OF 419 COMPOSED OF RX(73), RX(84), RX(85), RX(22)
RX(284) **CY** + **EQ** + EP + AI ===> **BD**



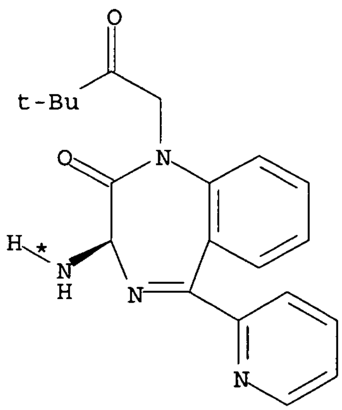
CY



EQ

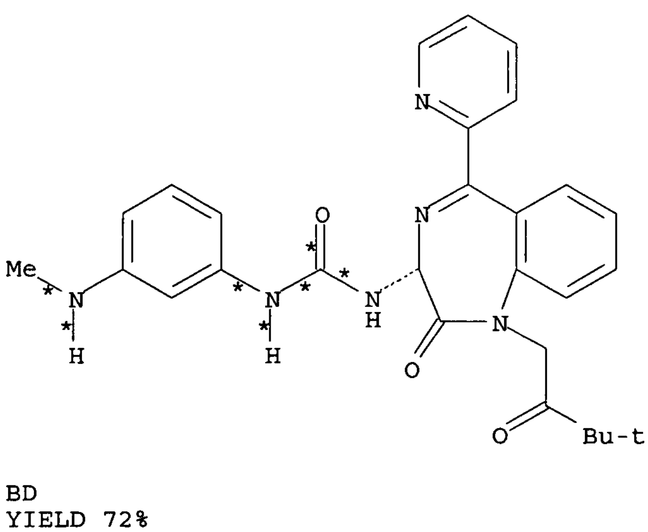


EP



AI

4 STEPS ⟶

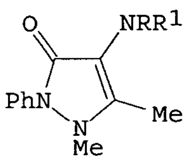


BD
YIELD 72%

```
RX(73)    RCT  CY 99-05-8

            STAGE(1)
               RGT  CD 1310-58-3 KOH
               SOL  123-91-1 Dioxane, 7732-18-5 Water

            STAGE(2)
               RCT  EQ 24424-99-5

          PRO  AR 111331-82-9

RX(84)    RCT  AR 111331-82-9

            STAGE(1)
               RGT  BQ 7646-69-7 NaH
               SOL  68-12-2 DMF

            STAGE(2)
               RCT  EP 74-88-4

            STAGE(3)
               RGT  FN 1310-65-2 LiOH
               SOL  67-56-1 MeOH

            STAGE(4)
               RGT  U 7647-01-0 HCl
               SOL  7732-18-5 Water

          PRO  FM 168162-30-9

RX(85)    RCT  FM 168162-30-9

            STAGE(1)
               RGT  G 121-44-8 Et3N, EK 541-41-3 ClCO2Et
               SOL  67-64-1 Me2CO

            STAGE(2)
               RGT  EL 26628-22-8 NaN3
               SOL  7732-18-5 Water

          PRO  BC 168162-32-1

RX(22)    RCT  BC 168162-32-1, AI 164343-31-1

            STAGE(1)
               SOL  75-09-2 CH2Cl2

            STAGE(2)
               RGT  U 7647-01-0 HCl
               SOL  7732-18-5 Water

            STAGE(3)
               RGT  O 1310-73-2 NaOH
               SOL  7732-18-5 Water

          PRO  BD 155488-25-8
          NTE  stereoselective
```

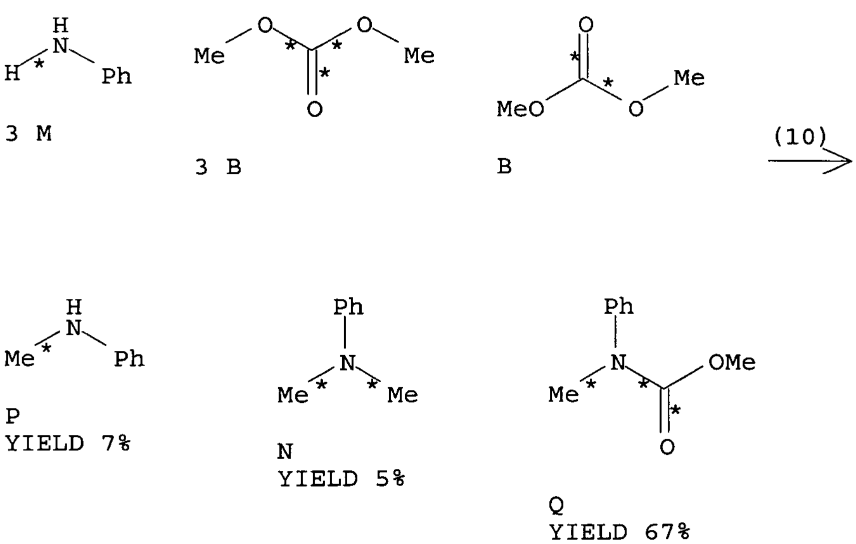
L60 ANSWER 37 OF 44 CASREACT

ACCESSION NUMBER: 112:178775 CASREACT
TITLE: Reactions with dimethyl carbonate. Part 3. Applications and mechanism of mono- or dimethylation of aromatic amines with dimethyl carbonate
AUTHOR(S): Lissel, Manfred; Rohani-Dezfuli, Ali Reza; Vogt, Gabriele
CORPORATE SOURCE: Fak. Chem., Univ. Bielefeld, Bielefeld, D-4800/1, Fed. Rep. Ger.
SOURCE: Journal of Chemical Research, Synopses (1989), (10), 312
CODEN: JRPSDC; ISSN: 0308-2342
DOCUMENT TYPE: Journal
LANGUAGE: English
GI


I

AB Methylation of aminoantipyrine I (R = R1 = H) with di-Me carbonate (II) in the presence of K2CO3 and 18-crown-6 at 90°, gave I (R = Me, R1 = CO2Me), which on hydrolysis with 10% KOH gave I (R = Me, R1 = H). At 50°, I (R = R1 = H) reacted with II in the presence of NaH, and 18-crown-6 to give I (R = H, R1 = CO2Me). Other aromatic amines, e.g., 4-R2C6H4NH2 (R2 = H, Me, Cl), were also N-methylated with II. The mechanism of the reactions is discussed.

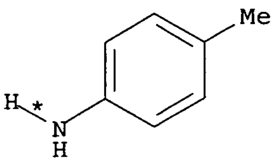
RX(10) OF 21 3 **M** + 4 **B** ===> **P** + N + Q



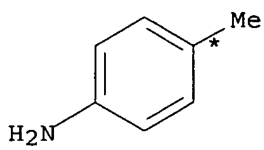
RX(10) RCT M **62-53-3**, B **616-38-6**
RGT D 584-08-7 K2CO3

PRO P **100-61-8**, N 121-69-7, Q 28685-60-1
CAT 17455-13-9 18-Crown-6
SOL 616-38-6 Me2CO3
NTE product varies with reaction conditions

RX(11) OF 21 8 **R** ===> **S** + T + U

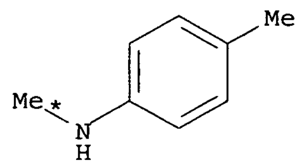


3 R

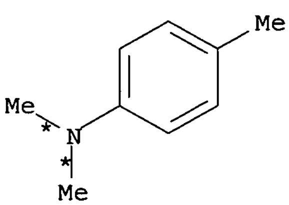


5 R

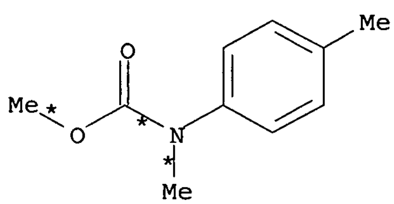
(11) →



S
YIELD 28%



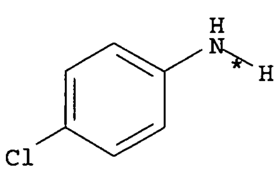
T
YIELD 45%



U
YIELD 27%

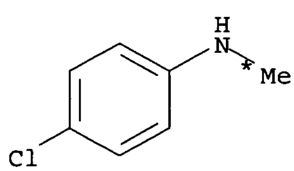
RX(11) RCT R **106-49-0**
RGT B **616-38-6** Me2CO3
PRO S **623-08-5**, T 99-97-8, U 121825-89-6
SOL 616-38-6 Me2CO3

RX(12) OF 21 2 **V** ===> **W** + X

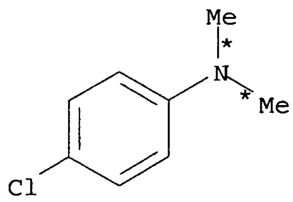


2 V

(12) →



W
YIELD 24%



X
YIELD 12%

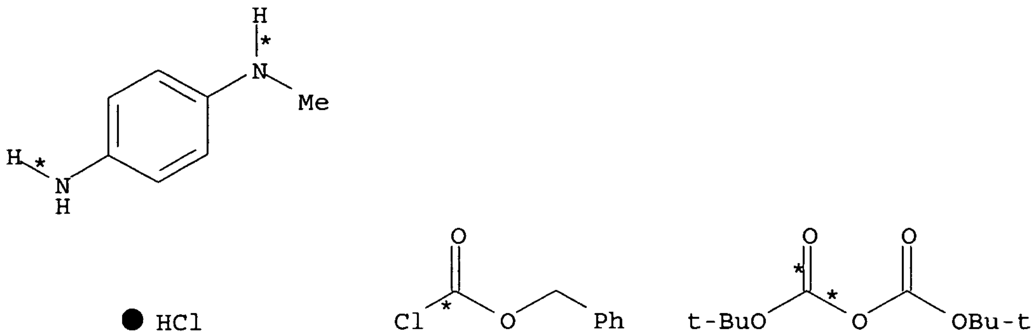
RX(12) RCT V **106-47-8**
RGT B **616-38-6** Me2CO3
PRO W **932-96-7**, X 698-69-1
SOL 616-38-6 Me2CO3

L60 ANSWER 38 OF 44 CASREACT

ACCESSION NUMBER: 110:75136 CASREACT
TITLE: Selective protection of polyamines: Synthesis of model compounds and spermidine derivatives
AUTHOR(S): Almeida, M. Lurdes S.; Grehn, Leif; Ragnarsson, Ulf
CORPORATE SOURCE: Biomed. Cent., Univ. Uppsala, Uppsala, S-751 23, Swed.
SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1988), (7), 1905-11
CODEN: JCPRB4; ISSN: 0300-922X
DOCUMENT TYPE: Journal
LANGUAGE: English

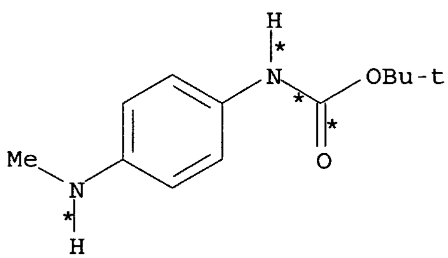
AB A general procedure for the selective protection of mixed primary-secondary (poly)amines, based on tert-butoxycarbonylation of carbamate groups (exhaustive tert-butoxycarbonylation) derived from the primary amino functions only, is reported. In most cases, benzyl (poly)carbamates are used for this purpose. Subsequent removal of all benzyloxycarbonyl (Z) groups from the resulting intermediates by catalytic hydrogenolysis liberates the secondary amino functions, while tert-butoxycarbonyl (Boc) is retained on the primary ones. Alternatively, selective removal of Z only from amino functions protected by both Z and Boc, which can be accomplished by base-catalyzed methanolysis, results in protected (poly)amines with Boc and Z on their primary and secondary amino groups, resp. The new reactions have been studied with two unsym. derivs. of ethylenediamine and p-phenylenediamine as model substances. The yields of most intermediates and the products were high. Addnl. expts. performed with spermidine gave N1,N8-Boc2-spermidine. The synthesis of N8-Boc-N1-Z-spermidine by the same approach is presented.

RX(60) OF 65 COMPOSED OF RX(18), RX(19), RX(20)
RX(60) **AL** + 2 B + **H** ===> **AO**



AL 2 B H

3
STEPS
$\longrightarrow$



AO
YIELD 98%

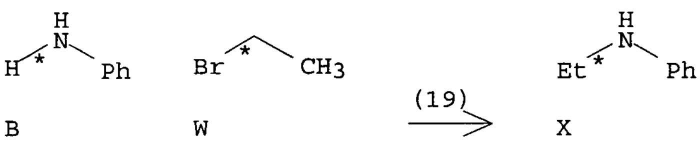
```
RX(18)     RCT  AL 118734-05-7, B 501-53-1
           RGT  AF 110-86-1 Pyridine
           PRO  AM 113283-90-2
           SOL  110-86-1 Pyridine

RX(19)     RCT  AM 113283-90-2, H 24424-99-5
           RGT  J 1122-58-3 4-DMAP
           PRO  AN 113283-92-4
           SOL  75-05-8 MeCN

RX(20)     RCT  AN 113283-92-4
           RGT  AP 1333-74-0 H2
           PRO  AO 113283-94-6
           CAT  7440-05-3 Pd
           SOL  67-56-1 MeOH
```

L60 ANSWER 39 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 110:172770 CASREACT
TITLE: Selective monoalkylation of amines
AUTHOR(S): Dalla Croce, Piero; La Rosa, Concetta; Ritieni,

Alberto
CORPORATE SOURCE: Dip. Chim. Org. Ind., Cent. CNR, Milan, 20133, Italy
SOURCE: Journal of Chemical Research, Synopses (1988), (10), 346-7
CODEN: JRPSDC; ISSN: 0308-2342
DOCUMENT TYPE: Journal
LANGUAGE: English

AB A simple method for the monoalkylation of primary amines is achieved by using N-tert-butoxycarbonyl (N-Boc) group as a protecting group. The reaction of RNH2 (R = Ph, substituted Ph, PhCH2, cyclohexyl, Bu) with di-tert-Bu dicarbonate gave the N-Boc derivative in almost quant. yield. Treatment to the N-Boc derivs. with NaH, followed by alkyl halide and then hydrolysis of the product with 10% aqueous HCl gave RNHR1 (R1 = Et, Me, Pr, Bu, PHCH2, allyl).

RX(19) OF 40 **B** + W ===> **X**



B W X
YIELD 95%

RX(19) RCT B **62-53-3**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

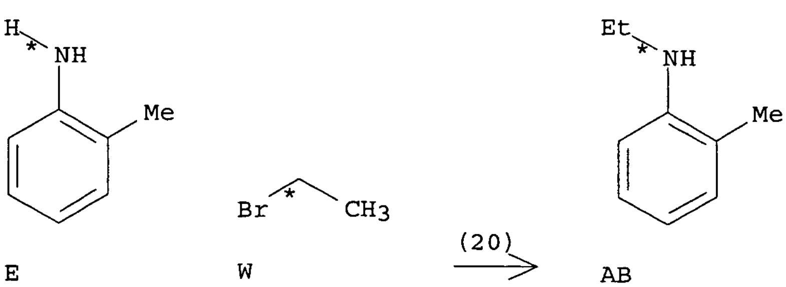
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO X **103-69-5**

RX(20) OF 40 **E** + W ===> **AB**



E W AB

RX(20) RCT E **95-53-4**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

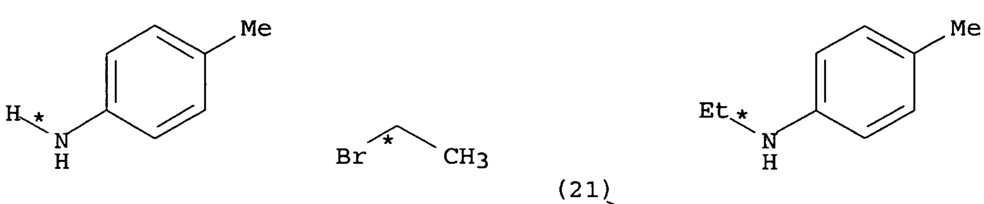
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AB **94-68-8**

RX(21) OF 40 **G** + W ===> **AC**



G W AC
YIELD 85%

RX(21) RCT G **106-49-0**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

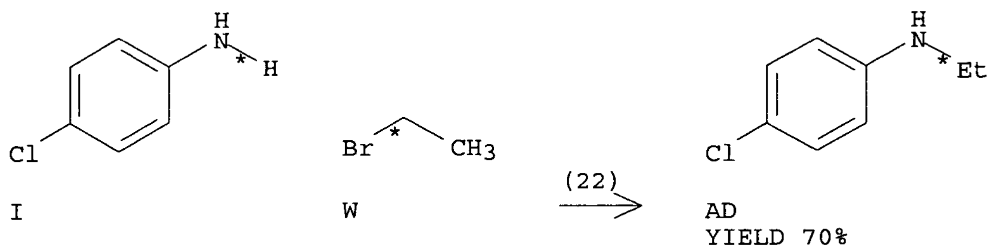
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AC **622-57-1**

RX(22) OF 40 **I** + W ===> **AD**



RX(22) RCT I **106-47-8**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

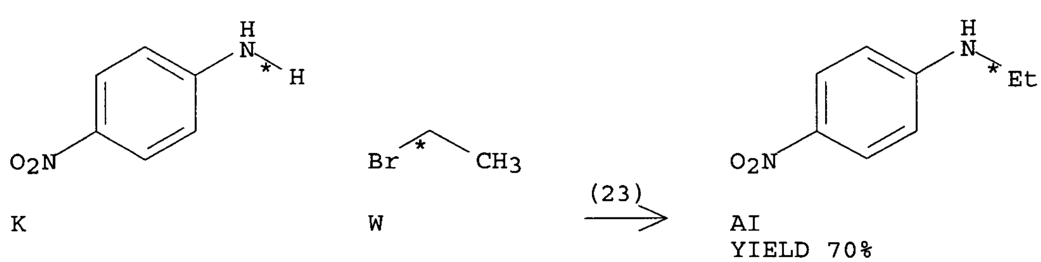
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AD **13519-75-0**

RX(23) OF 40 **K** + W ===> **AI**



RX(23) RCT K **100-01-6**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

STAGE(2)
RGT Y 7646-69-7 NaH

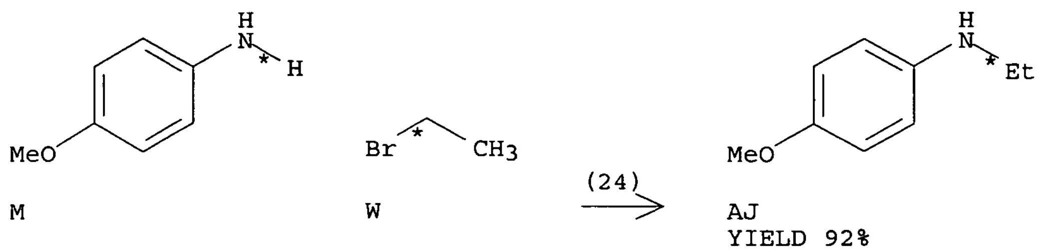
STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl

SOL 7732-18-5 Water

PRO AI **3665-80-3**

RX(24) OF 40 **M** + W ===> **AJ**



RX(24) RCT M **104-94-9**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

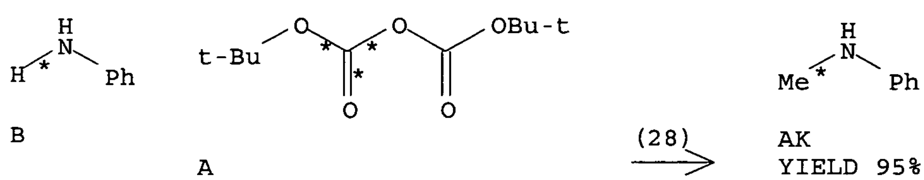
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT W 74-96-4

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AJ **104-48-3**

RX(28) OF 40 **B** + **A** ===> **AK**



RX(28) RCT B **62-53-3**, A **24424-99-5**

STAGE(1)
SOL 109-99-9 THF

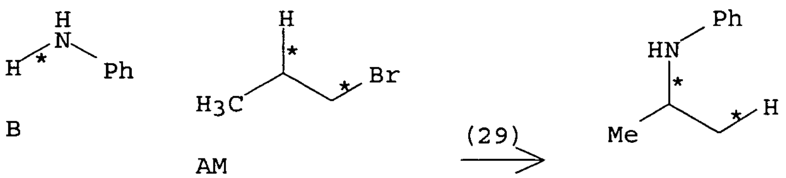
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RGT AL 74-88-4 MeI

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AK **100-61-8**

RX(29) OF 40 **B** + AM ===> **AN**



B

AM

AN
YIELD 30%

RX(29) RCT B **62-53-3**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

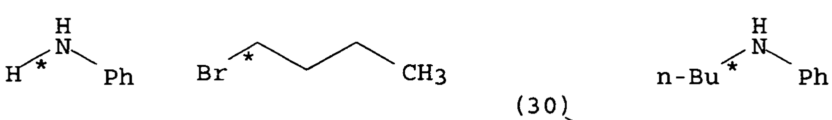
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT AM 106-94-5

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AN **768-52-5**

RX(30) OF 40 **B** + AO ===> **AP**



B

AO

AP
YIELD 75%

RX(30) RCT B **62-53-3**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

STAGE(2)
RGT Y 7646-69-7 NaH

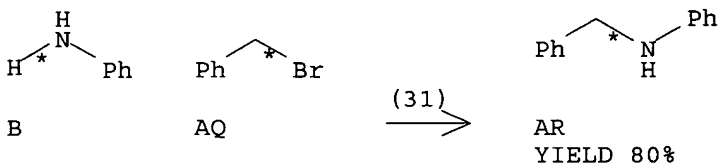
STAGE(3)

RCT AO 109-65-9

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AP **1126-78-9**

RX(31) OF 40 **B** + AQ ===> **AR**



RX(31) RCT B **62-53-3**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

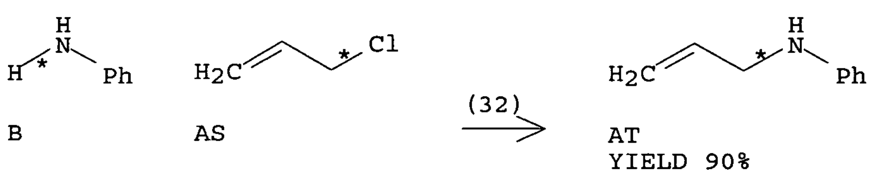
STAGE(2)
RGT Y 7646-69-7 NaH

STAGE(3)
RCT AQ 100-39-0

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AR **103-32-2**

RX(32) OF 40 **B** + AS ===> **AT**



RX(32) RCT B **62-53-3**

STAGE(1)
RGT A **24424-99-5** (Boc)2O
SOL 109-99-9 THF

STAGE(2)
RGT Y 7646-69-7 NaH

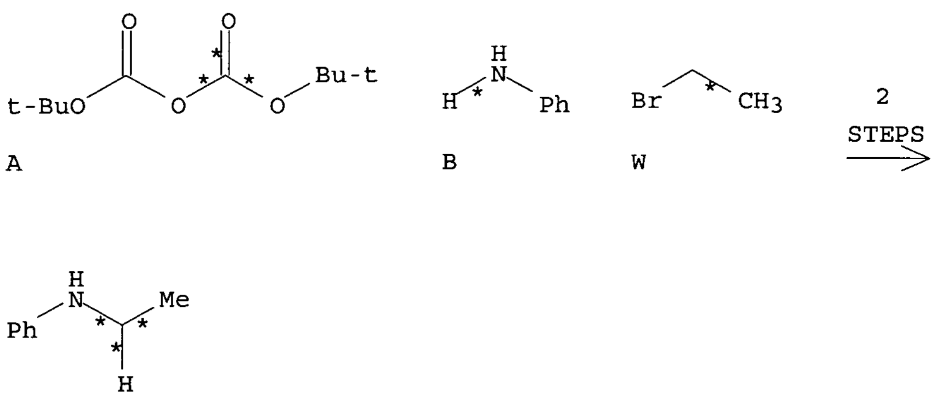
STAGE(3)

RCT AS 107-05-1

STAGE(4)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AT **589-09-3**
NTE Chloride assumed

RX(33) OF 40 COMPOSED OF RX(1), RX(11)
RX(33) **A** + **B** + W ===> **X**



X
YIELD 98%

RX(1) RCT A **24424-99-5**, B **62-53-3**
PRO C 3422-01-3
SOL 109-99-9 THF

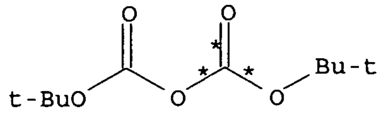
RX(11) RCT C 3422-01-3

STAGE(1)
RGT Y 7646-69-7 NaH
SOL 109-99-9 THF

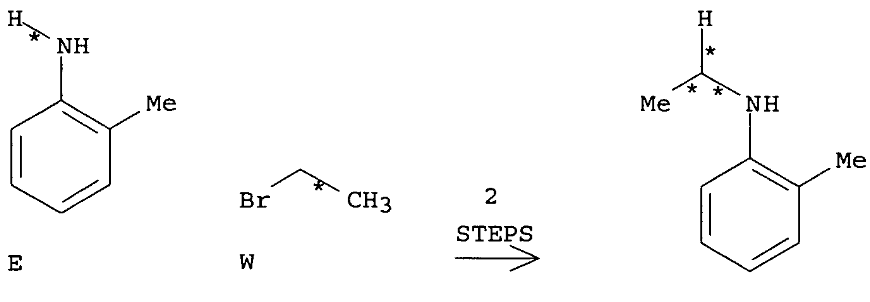
STAGE(2)
RCT W 74-96-4
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO X **103-69-5**
NTE Dioxane may also be used as solvent

RX(34) OF 40 COMPOSED OF RX(2), RX(12)
RX(34) **A** + **E** + W ===> **AB**



A



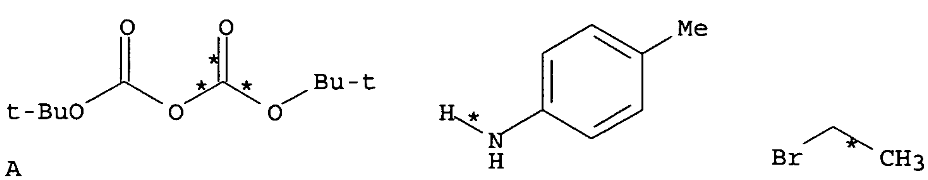
E W AB

YIELD 82%

```
RX(2)       RCT  A 24424-99-5, E 95-53-4
            PRO  F 74965-31-4
            SOL  109-99-9 THF

RX(12)      RCT  F 74965-31-4

              STAGE(1)
                 RGT  Y 7646-69-7 NaH
                 SOL  109-99-9 THF

              STAGE(2)
                 RCT  W 74-96-4

              STAGE(3)
                 RGT  Z 7647-01-0 HCl
                 SOL  7732-18-5 Water

            PRO  AB 94-68-8
            NTE  Dioxane may also be used as solvent
```

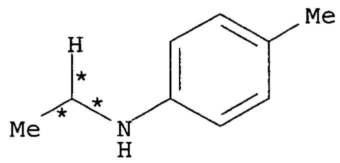
RX(35) OF 40 COMPOSED OF RX(3), RX(13)

RX(35) **A** + **G** + **W** ===> **AC**



A G W

2 STEPS →



AC
YIELD 91%

RX(3) RCT A **24424-99-5**, G **106-49-0**
PRO H 14618-59-8
SOL 109-99-9 THF

RX(13) RCT H 14618-59-8

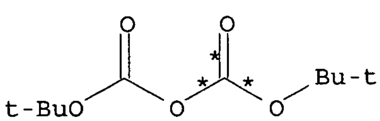
STAGE(1)
RGT Y 7646-69-7 NaH
SOL 109-99-9 THF

STAGE(2)
RCT W 74-96-4

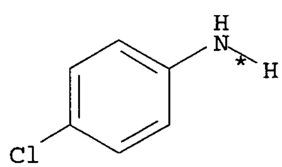
STAGE(3)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AC **622-57-1**
NTE Dioxane may also be used as solvent

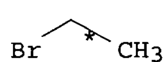
RX(36) OF 40 COMPOSED OF RX(4), RX(14)
RX(36) **A** + **I** + W ===> **AD**



A

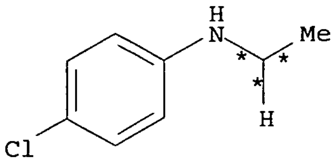


I



W

2 STEPS →



AD
YIELD 73%

RX(4) RCT A **24424-99-5**, I **106-47-8**
PRO J 18437-66-6
SOL 109-99-9 THF

RX(14) RCT J 18437-66-6

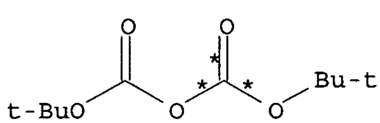
STAGE(1)
RGT Y 7646-69-7 NaH
SOL 109-99-9 THF

STAGE(2)
RCT W 74-96-4

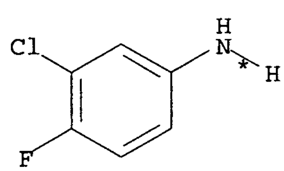
STAGE(3)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

PRO AD **13519-75-0**
NTE Dioxane may also be used as solvent

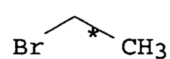
RX(37) OF 40 COMPOSED OF RX(7), RX(15)
RX(37) **A** + **O** + W ===> **AE**



A

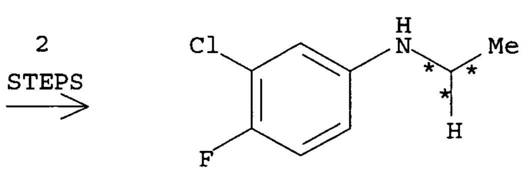


O



W

2
STEPS



AE
YIELD 87%

RX(7) RCT A **24424-99-5**, O **367-21-5**
PRO P 119951-96-1
SOL 109-99-9 THF

RX(15) RCT P 119951-96-1

STAGE(1)
RGT Y 7646-69-7 NaH
SOL 109-99-9 THF

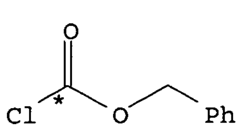
STAGE(2)
RCT W 74-96-4

STAGE(3)
RGT Z 7647-01-0 HCl
SOL 7732-18-5 Water

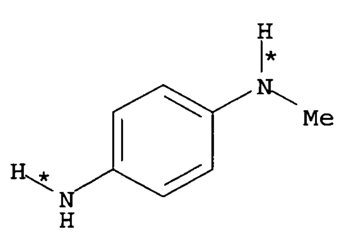
PRO AE **106847-36-3**
NTE Dioxane may also be used as solvent

L60 ANSWER 40 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 108:112816 CASREACT
TITLE: Selective protection of mixed primary-secondary amines. Simple preparation of N1,N8-bis(tert-butyloxycarbonyl)spermidine
AUTHOR(S): Almeida, M. Lurdes S.; Grehn, Leif; Ragnarsson, Ulf
CORPORATE SOURCE: Inst. Biochem., Univ. Uppsala, Uppsala, S-751 23, Swed.
SOURCE: Journal of the Chemical Society, Chemical Communications (1987), (16), 1250-1
CODEN: JCCCAT; ISSN: 0022-4936
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A procedure for the selective protection of mixed primary-secondary amines, based on acylation followed by exhaustive tert-butoxycarbonylation is presented. Thus, H2N(CH2)3NH(CH2)4NH2 was treated with PhCH2O2CCl and aqueous Na2CO3, followed by (Me3CO)2CO and 4-(dimethylamino)pyridine to give RR1N(CH2)3NR2(CH2)4NRR1 (I, R = R2 = PhCH2O2C, R1 = Me3CO2C). Transfer hydrogenation of the latter compound using HCO2NH4 and Pd-C in MeOH gave I (R = R2 = H, R1 = Me3CO2C), whereas, treatment with Me2NC(:NH)NMe2 in MeOH gave I (R = H, R1 = Me3CO2C, R2 = PhCH2O2C).

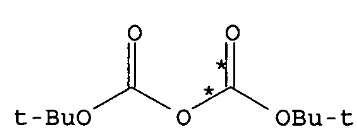
RX(29) OF 34 COMPOSED OF RX(3), RX(6), RX(8)
RX(29) 2 A + **G** + **I** ===> **R**



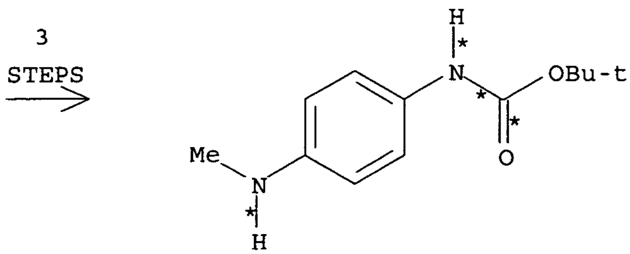
2 A



G



I

3
STEPS


R
YIELD 98%

RX(3) RCT A 501-53-1, G **623-09-6**
RGT D 110-86-1 Pyridine
PRO H 113283-90-2

RX(6) RCT I **24424-99-5**, H 113283-90-2
RGT K 1122-58-3 4-DMAP
PRO M 113283-92-4
SOL 75-05-8 MeCN

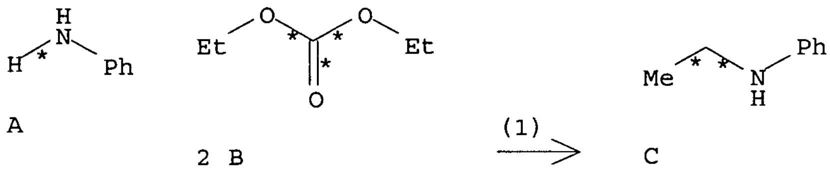
RX(8) RCT M 113283-92-4
RGT S 1333-74-0 H2
PRO R **113283-94-6**
CAT 7440-05-3 Pd
SOL 67-56-1 MeOH

L60 ANSWER 41 OF 44 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 104:224725 CASREACT
TITLE: Carbamates
INVENTOR(S): Frulla, Floro F.; Stuber, Fred A.; Whitman, Peter J.
PATENT ASSIGNEE(S): Dow Chemical Co., USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4550188	A	19851029	US 1984-625060	19840627
PRIORITY APPLN. INFO.:			US 1984-625060	19840627

AB Carbamates useful as precursors for insecticides and as intermediates for organic mono- and polyisocyanates (no data) are manufactured at low temps. and in high yields by heating an organic carbonate, i.e. a dialkyl, diaryl, diaralkyl, or cyclic carbonate ester, with an aromatic primary amine at 75-200° in the presence of Al metal catalyst with a promoter containing a Hg salt compound and iodine. Thus, a flask containing aniline 7.5, di-Et carbonate 96.2, Al foil 0.5, iodine 0.01, and HgCl2 0.01 g was heated at 130° for 17.5 h, diluted with 200 mL H2O, and extracted to give 11.6 g residue containing 92.4% Et N-phenylcarbamate and 0.7% diphenylurea. Similarly, CH2(C6H4NHCO2Et)2 was prepared in 85.8% yield.

RX(1) OF 2 **A** + 2 **B** ===> **C**



RX(1) RCT A **62-53-3**, B **105-58-8**
PRO C **103-69-5**

L60 ANSWER 42 OF 44 CAPLUS

ACCESSION NUMBER: 2006:117009 CAPLUS
DOCUMENT NUMBER: 144:212531
TITLE: Catalytic method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines
INVENTOR(S): Russ, Werner Hubert; Hutchings, Michael; Ebenezer, Warren James
PATENT ASSIGNEE(S): Dystar Textilfarben G.m.b.H. & Co. Deutschland K.-G., Germany
SOURCE: PCT Int. Appl., 12 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2006013164	A1	20060209	WO 2005-EP53591	20050722

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 102004036787	A1	20060323	DE 2004-102004036787	20040729
PRIORITY APPLN. INFO.:			DE 2004-102004036787A	20040729

OTHER SOURCE(S): MARPAT 144:212531

AB Monoalkylated aromatic diamines [e.g., 2-(methylamino)aniline] are prepared by the monoalkylation of a (hetero)aromatic diamine (e.g., o-phenylenediamine) with a cyclic or diorganyl **carbonate** (e.g., di-Me **carbonate**) in the presence of a zeolite catalyst (e.g., NaY-faujasite-type zeolite).

IC ICM C07C209-00
ICS C07C211-51

CC 25-4 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)

Section cross-reference(s): 45, 67
IT **Faujasite**-type zeolites
RL: CAT (Catalyst use); USES (Uses)
(NaY-**faujasite**; catalysts in a method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT Zeolites (synthetic), uses
RL: CAT (Catalyst use); USES (Uses)
(catalysts in a method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT **Carbonates**, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(catalytic method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT **Amines, preparation**
RL: SPN (Synthetic preparation); PREP (Preparation)
(diamines, **aromatic**, monoalkylated; catalytic method for the production of monoalkylated (hetero)**aromatic** diamines using **carbonates** and (hetero)aryl diamines)
IT Alkylation
(monoalkylation; catalytic method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT Alkylation catalysts
(monoalkylation; zeolites in a method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT 554-13-2, Lithium **carbonate** 5006-97-3, Lithium **bicarbonate**
RL: CAT (Catalyst use); USES (Uses)
(catalysts in a method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT 95-54-5, o-Phenylenediamine, reactions 96-49-1, Ethylene **carbonate** 616-38-6, Dimethyl **carbonate**
RL: RCT (Reactant); RACT (Reactant or reagent)
(catalytic method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
IT 4760-34-3P, 2-(Methylamino)aniline
RL: SPN (Synthetic preparation); PREP (Preparation)
(catalytic method for the production of monoalkylated (hetero)aromatic diamines using **carbonates** and (hetero)aryl diamines)
REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L60 ANSWER 43 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1995:376454 CAPLUS
DOCUMENT NUMBER: 122:164029
TITLE: Selectivity to N-mono or dialkylation in the reaction of aniline with dimethyl **carbonate** on **faujasite**, EMT and beta alkaline zeolites
AUTHOR(S): Rao, P. R. Hari Prasad; Massiani, Pascale; Barthomeuf, Denise
CORPORATE SOURCE: Lab. Reactivite Surface, Univ. Pierre Marie Curie, Paris, 75252, Fr.
SOURCE: Catalysis Letters (1995), 31(1), 115-20
CODEN: CALEER; ISSN: 1011-372X
PUBLISHER: Baltzer
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Zeolite K-EMT is as active and selective to N-monomethylation as KY in the

alkylation of aniline with di-Me **carbonate** at 408 or 453 K. No C-alkylation occurs in the temperature range studied. At 503 K, KX and CsX are about 75% selective for dialkylation to N,N-dimethylaniline with about 97% conversion.

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)
Section cross-reference(s): 67

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(Cs EMT-type; selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(Cs beta; selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(K EMT-type; selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Alkylation
Alkylation catalysts
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(CsX, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(CsY, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(H beta, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

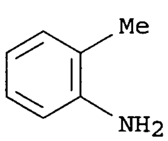
IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(K beta, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(KX, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

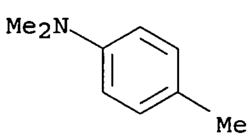
IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(KY, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(Na EMT-type, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline

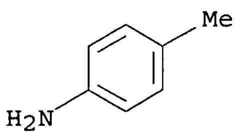
zeolites)
IT Zeolites, uses
RL: CAT (Catalyst use); USES (Uses)
(Na beta, selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)
IT **95-53-4P**, o-Toluidine, preparation **99-97-8P**, N,N-Dimethyl-p-toluidine **106-49-0P**, p-Toluidine, preparation
RL: BYP (Byproduct); **PREP (Preparation)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)
IT **100-61-8P**, N-Methylaniline, preparation **121-69-7P**, N,N-Dimethylaniline, preparation
RL: IMF (Industrial manufacture); **PREP (Preparation)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)
IT **62-53-3**, Aniline, reactions 616-38-6, Dimethyl **carbonate**
RL: PEP (Physical, engineering or chemical process); **RCT (Reactant)**; PROC (Process); **RACT (Reactant or reagent)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)
IT **95-53-4P**, o-Toluidine, preparation **99-97-8P**, N,N-Dimethyl-p-toluidine **106-49-0P**, p-Toluidine, preparation
RL: BYP (Byproduct); **PREP (Preparation)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)
RN 95-53-4 CAPLUS
CN Benzenamine, 2-methyl- (9CI) (CA INDEX NAME)



RN 99-97-8 CAPLUS
CN Benzenamine, N,N,4-trimethyl- (9CI) (CA INDEX NAME)



RN 106-49-0 CAPLUS
CN Benzenamine, 4-methyl- (9CI) (CA INDEX NAME)



IT **100-61-8P**, N-Methylaniline, preparation **121-69-7P**,
N,N-Dimethylaniline, preparation
RL: IMF (Industrial manufacture); **PREP (Preparation)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

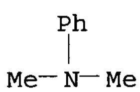
RN 100-61-8 CAPLUS

CN Benzenamine, N-methyl- (9CI) (CA INDEX NAME)

Me— NH— Ph

RN 121-69-7 CAPLUS

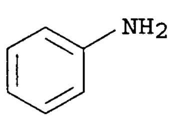
CN Benzenamine, N,N-dimethyl- (9CI) (CA INDEX NAME)



IT **62-53-3**, Aniline, reactions
RL: PEP (Physical, engineering or chemical process); **RCT (Reactant)**; PROC (Process); **RACT (Reactant or reagent)**
(selectivity to N-mono- or dialkylation of aniline with di-Me **carbonate** over **faujasite**, EMT, and beta alkaline zeolites)

RN 62-53-3 CAPLUS

CN Benzenamine (9CI) (CA INDEX NAME)



L60 ANSWER 44 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:	1993:519064 CAPLUS
DOCUMENT NUMBER:	119:119064
TITLE:	Process for reclaiming a thermoset polymer
INVENTOR(S):	Jones, C. Andrew; Gaffney, Anne M.; Leyshon, David W.; Cozzone, Glenn E.; Sofranko, John A.
PATENT ASSIGNEE(S):	ACRO Chemical Technology, L. P., USA
SOURCE:	U.S., 10 pp. CODEN: USXXAM
DOCUMENT TYPE:	Patent
LANGUAGE:	English
FAMILY ACC. NUM. COUNT:	1
PATENT INFORMATION:	

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5192809	A	19930309	US 1992-860638	19920330

PRIORITY APPLN. INFO.: US 1992-860638 19920330

AB Filled thermosetting polymer waste is reclaimed by contacting polymer and particulate zeolite-containing catalyst in a fluidized bed at a temperature to give a coarse filler, coke, a volatile organic matter and a spent catalyst. The first stream comprising the volatile organic matter is withdrawn from the reaction zone, and then a second stream containing the other components; the second stream is heated in presence of O_2 to convert coke to CO_2 and H_2O and to regenerate catalyst, which is separated along with filler. Thus, volatile orgs. were recovered by heating glass fiber-filled maleic anhydride-styrene copolymer at 21-681° in presence of zeolite-containing catalyst; mass balance was 96% and O_2 balance was 100%.

IC ICM C08L101-00

INCL 521040000

CC 38-3 (Plastics Fabrication and Uses)

IT 12173-28-3, **Faujasite** 12173-98-7, Mordenite 12417-81-1, Offretite 12510-42-8, Erionite
RL: CAT (Catalyst use); USES (Uses)
(catalysts, for reclamation of volatile orgs. from thermosets)

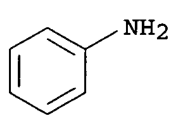
IT **62-53-3P**, Aniline, uses **100-41-4P**, Ethylbenzene, uses
RL: **PREP (Preparation)**; USES (Uses)
(reclamation recovery of, from thermosets, using zeolite catalysts)

IT 471-34-1, Calcium **carbonate**, uses
RL: USES (Uses)
(thermosets filled by, reclamation of, using zeolite catalysts)

IT **62-53-3P**, Aniline, uses **100-41-4P**, Ethylbenzene, uses
RL: **PREP (Preparation)**; USES (Uses)
(reclamation recovery of, from thermosets, using zeolite catalysts)

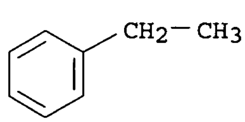
RN 62-53-3 CAPLUS

CN Benzenamine (9CI) (CA INDEX NAME)



RN 100-41-4 CAPLUS

CN Benzene, ethyl- (7CI, 8CI, 9CI) (CA INDEX NAME)



```
=> file casreact
FILE 'CASREACT' ENTERED AT 16:00:03 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is
held by the publishers listed in the PUBLISHER (PB) field (available
for records published or updated in Chemical Abstracts after December
26, 1996), unless otherwise indicated in the original publications.

FILE CONTENT:1840 - 30 Apr 2006 VOL 144 ISS 18

New CAS Information Use Policies, enter HELP USAGETERMS for details.

************************************************************
*                                                          *
*     CASREACT now has more than 10 million reactions      *
*                                                          *
************************************************************

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991)
provided by InfoChem, INPI data prior to 1986, and Biotransformations
database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance
identification.

=> d stat que L39
L5                STR
```

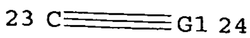
C 30 N 31

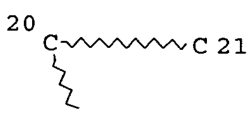
25 C M3

26 C M2

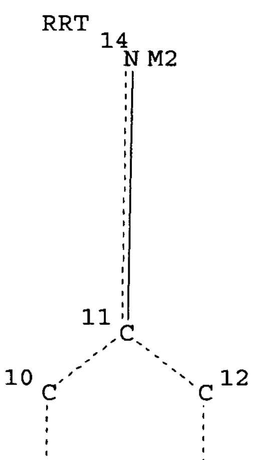
27 C M1

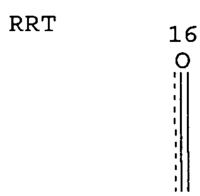
Page 1-A



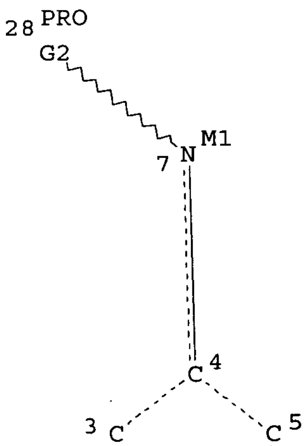


Page 1-B

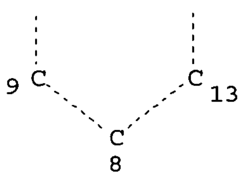


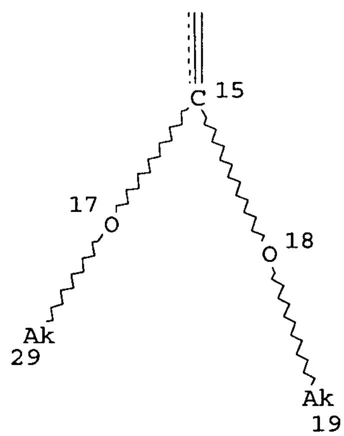


Page 2-A

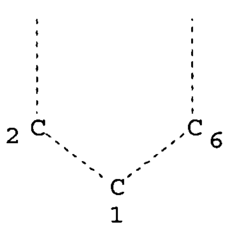


Page 2-B



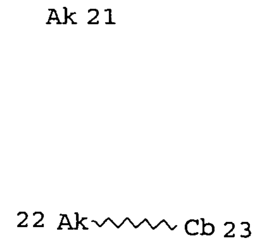


Page 3-A

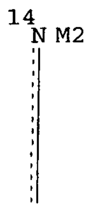


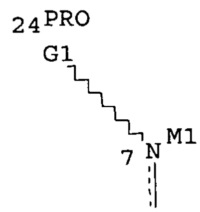
Page 3-B

```
VAR G1=30/31
VAR G2=20/23/25/26/27
NODE ATTRIBUTES:
HCOUNT   IS M1      AT   7
HCOUNT   IS M2      AT  14
HCOUNT   IS M3      AT  25
HCOUNT   IS M2      AT  26
HCOUNT   IS M1      AT  27
NSPEC    IS R       AT   1
NSPEC    IS R       AT   2
NSPEC    IS R       AT   3
NSPEC    IS R       AT   4
NSPEC    IS R       AT   5
NSPEC    IS R       AT   6
NSPEC    IS C       AT   7
NSPEC    IS R       AT   8
NSPEC    IS R       AT   9
NSPEC    IS R       AT  10
NSPEC    IS R       AT  11
NSPEC    IS R       AT  12
NSPEC    IS R       AT  13
NSPEC    IS C       AT  14
NSPEC    IS C       AT  15
NSPEC    IS C       AT  16
NSPEC    IS C       AT  17
NSPEC    IS C       AT  18
NSPEC    IS C       AT  19
NSPEC    IS C       AT  20
NSPEC    IS C       AT  21
NSPEC    IS C       AT  22
NSPEC    IS C       AT  23
NSPEC    IS C       AT  24
NSPEC    IS C       AT  25
NSPEC    IS C       AT  26
NSPEC    IS C       AT  27
NSPEC    IS C       AT  28
NSPEC    IS C       AT  29
DEFAULT MLEVEL IS ATOM
MLEVEL   IS CLASS   AT   7 14 15 16 17 18 19 20 21 22 23 25 26 27 29 30 31
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  31

STEREO ATTRIBUTES: NONE
L29           91 SEA FILE=CASREACT SSS FUL L5 (    702 REACTIONS)
L34              STR
```

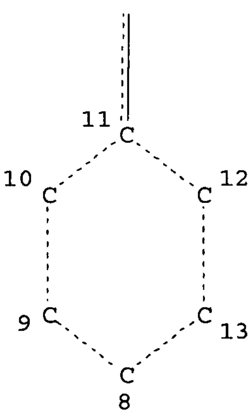


RRT

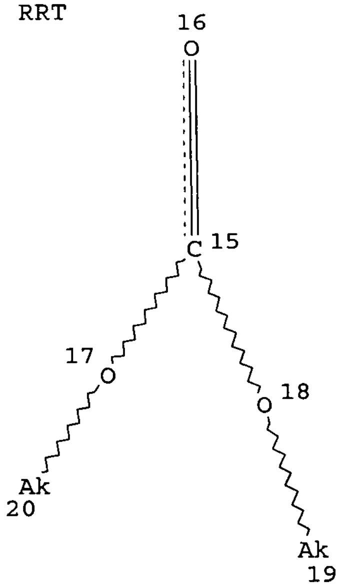


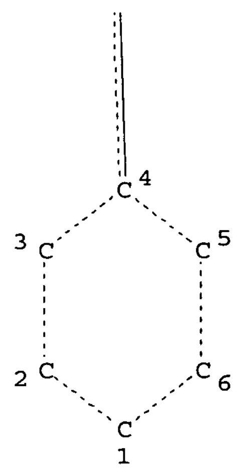


Page 1-A



RRT





Page 2-A
VAR G1=21/22
NODE ATTRIBUTES:

```
HCOUNT  IS M1      AT   7
HCOUNT  IS M2      AT  14
NSPEC   IS R       AT   1
NSPEC   IS R       AT   2
NSPEC   IS R       AT   3
NSPEC   IS R       AT   4
NSPEC   IS R       AT   5
NSPEC   IS R       AT   6
NSPEC   IS C       AT   7
NSPEC   IS R       AT   8
NSPEC   IS R       AT   9
NSPEC   IS R       AT  10
NSPEC   IS R       AT  11
NSPEC   IS R       AT  12
NSPEC   IS R       AT  13
NSPEC   IS C       AT  14
NSPEC   IS C       AT  15
NSPEC   IS C       AT  16
NSPEC   IS C       AT  17
NSPEC   IS C       AT  18
NSPEC   IS C       AT  19
NSPEC   IS C       AT  20
NSPEC   IS C       AT  21
NSPEC   IS C       AT  22
NSPEC   IS C       AT  23
NSPEC   IS C       AT  24
CONNECT IS E1   RC AT  21
CONNECT IS E2   RC AT  22
DEFAULT MLEVEL IS ATOM
MLEVEL  IS CLASS  AT   7 14 15 16 17 18 19 20 21 22
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  24

STEREO ATTRIBUTES: NONE
L36           48 SEA FILE=CASREACT SUB=L29 SSS FUL L34 (    243 REACTIONS)
L37           32 SEA FILE=CASREACT ABB=ON  PLU=ON  SELVA M?/AU
L38           57 SEA FILE=CASREACT ABB=ON  PLU=ON  TUNDO P?/AU
L39            7 SEA FILE=CASREACT ABB=ON  PLU=ON  L36 AND (L37 OR L38)


=> file caplus
FILE 'CAPLUS' ENTERED AT 16:00:22 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)
```

```
FILE COVERS 1907 - 3 May 2006  VOL 144 ISS 19
FILE LAST UPDATED: 2 May 2006  (20060502/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply.
They are available for your review at:

http://www.cas.org/infopolicy.html
'OBI' IS DEFAULT SEARCH FIELD FOR 'CAPLUS' FILE

=> d stat que L57
L8          3769 SEA FILE=CAPLUS ABB=ON  PLU=ON  FAUJASIT?/OBI
L9             1 SEA FILE=REGISTRY ABB=ON  PLU=ON  ANILINE/CN
L10        49252 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9
L11           23 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L10
L12        25354 SEA FILE=CAPLUS ABB=ON  PLU=ON  L9 (L) (RCT OR RGT OR RACT)/RL

L13           16 SEA FILE=CAPLUS ABB=ON  PLU=ON  L8 AND L12
L14          297 SEA FILE=REGISTRY ABB=ON  PLU=ON  (62-53-3/BI OR 1335-30-4/BI
                 OR 100-61-8/BI OR 106-49-0/BI OR 121-69-7/BI OR 104-94-9/BI OR
                 95-53-4/BI OR 100-01-6/BI OR 106-47-8/BI OR 108-95-2/BI OR
                 616-38-6/BI OR 7631-86-9/BI OR 100-15-2/BI OR 108-44-1/BI OR
                 108-88-3/BI OR 1314-23-4/BI OR 1330-20-7/BI OR 1344-28-1/BI OR
                 67-56-1/BI OR 71-43-2/BI OR 99-97-8/BI OR 100-23-2/BI OR
                 100-41-4/BI OR 100-46-9/BI OR 101-77-9/BI OR 101-81-5/BI OR
                 103-32-2/BI OR 103-69-5/BI OR 108-67-8/BI OR 110-86-1/BI OR
                 12173-28-3/BI OR 12173-98-7/BI OR 1309-48-4/BI OR 1314-35-8/BI
                 OR 13463-67-7/BI OR 141814-27-9/BI OR 150-13-0/BI OR 23713-49-7
                 /BI OR 3459-92-5/BI OR 50-00-0/BI OR 588-59-0/BI OR 589-16-2/BI
                  OR 5961-59-1/BI OR 623-08-5/BI OR 643-28-7/BI OR 67-63-0/BI
                 OR 7440-05-3/BI OR 7440-06-4/BI OR 7440-50-8/BI OR 90-04-0/BI
                 OR 932-96-7/BI OR 98-82-8/BI OR 98-95-3/BI OR 100-00-5/BI OR
                 100-17-4/BI OR 100-42-5/BI OR 100-44-7/BI OR 100-47-0/BI OR
                 100-66-3/BI OR 10072-05-6/BI OR 102-07-8/BI OR 103-29-7/BI OR
                 103-65-1/BI OR 103-67-3/BI OR 103-73-1/BI OR 103-83-3/BI OR
                 104-13-2/BI OR 104-76-7/BI OR 10402-24-1/BI OR 105-05-5/BI OR
                 105-58-8/BI OR 10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR
                 106-98-9/BI OR 108-38-3/BI OR 108-39-4/BI OR 108-45-2/BI OR
                 108-47-4/BI OR 108-48-5/BI OR 108-89-4/BI OR 108-99-6/BI OR
                 109-06-8/BI OR 109-75-1/BI OR 110-54-3/BI OR 110-82-7/BI OR
                 11098-99-0/BI OR 11099-11-9/BI OR 111-77-3/BI OR 11116-47-5/BI
                 OR 11118-57-3/BI OR 112-49-2/BI OR 112-62-9/BI OR 115-07-1/BI
                 OR 115-11-7/BI OR 119-61-9/BI OR 119-65-3/BI OR 1197-19-9/BI
                 OR 120-12-7/BI OR 121-44-8/BI OR 1219-99-4/BI OR 122-39-4/BI
                 OR 1227-44-7/BI OR 12417-81-1/BI OR 12510-42-8/BI OR 12627-00-8
                 /BI OR 12737-86-9/BI OR 129-00-0/BI OR 1300-71-6/BI OR
                 1300-73-8/BI OR 1302-42-7/BI OR 1303-86-2/BI OR 1305-78-8/BI
                 OR 1308-38-9/BI OR 1310-73-2/BI OR 1311-10-0/BI OR
L16       310605 SEA FILE=CAPLUS ABB=ON  PLU=ON  L14/PREP
L17           20 SEA FILE=CAPLUS ABB=ON  PLU=ON  L16 AND L11
L18       447798 SEA FILE=CAPLUS ABB=ON  PLU=ON  ?CARBONAT?/BI
L19            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L11 AND L18
L21          218 SEA FILE=REGISTRY ABB=ON  PLU=ON  (1335-30-4/BI OR 62-53-3/BI
                 OR 100-61-8/BI OR 106-49-0/BI OR 121-69-7/BI OR 100-01-6/BI OR
                 104-94-9/BI OR 106-47-8/BI OR 616-38-6/BI OR 95-53-4/BI OR
                 100-15-2/BI OR 7631-86-9/BI OR 99-97-8/BI OR 100-23-2/BI OR
                 100-46-9/BI OR 101-77-9/BI OR 103-32-2/BI OR 108-44-1/BI OR
                 108-88-3/BI OR 110-86-1/BI OR 1309-48-4/BI OR 1314-23-4/BI OR
                 1314-35-8/BI OR 1330-20-7/BI OR 1344-28-1/BI OR 13463-67-7/BI
                 OR 141814-27-9/BI OR 3459-92-5/BI OR 50-00-0/BI OR 589-16-2/BI
                 OR 5961-59-1/BI OR 623-08-5/BI OR 643-28-7/BI OR 67-56-1/BI OR
```

```
               71-43-2/BI OR 7440-05-3/BI OR 7440-06-4/BI OR 7440-50-8/BI OR
               932-96-7/BI OR 100-41-4/BI OR 100-44-7/BI OR 100-47-0/BI OR
               100-66-3/BI OR 10072-05-6/BI OR 101-81-5/BI OR 102-07-8/BI OR
               103-65-1/BI OR 103-67-3/BI OR 103-69-5/BI OR 103-73-1/BI OR
               103-83-3/BI OR 104-13-2/BI OR 10402-24-1/BI OR 105-05-5/BI OR
               105-58-8/BI OR 10541-83-0/BI OR 106-42-3/BI OR 106-44-5/BI OR
               106-98-9/BI OR 108-38-3/BI OR 108-39-4/BI OR 108-67-8/BI OR
               108-89-4/BI OR 108-95-2/BI OR 109-75-1/BI OR 110-54-3/BI OR
               110-82-7/BI OR 11098-99-0/BI OR 11099-11-9/BI OR 111-77-3/BI
               OR 11118-57-3/BI OR 112-49-2/BI OR 115-07-1/BI OR 115-11-7/BI
               OR 119-65-3/BI OR 1197-19-9/BI OR 120-12-7/BI OR 121-44-8/BI
               OR 12173-28-3/BI OR 12173-98-7/BI OR 1219-99-4/BI OR 1227-44-7/
               BI OR 12627-00-8/BI OR 129-00-0/BI OR 1300-71-6/BI OR 1300-73-8
               /BI OR 1302-42-7/BI OR 1303-86-2/BI OR 1305-78-8/BI OR
               1308-38-9/BI OR 1310-73-2/BI OR 1311-10-0/BI OR 1314-13-2/BI
               OR 1314-56-3/BI OR 1314-62-1/BI OR 13308-51-5/BI OR 1333-41-1/B
               I OR 13330-29-5/BI OR 1345-13-7/BI OR 135-01-3/BI OR 13519-75-0
               /BI OR 13519-80-7/BI OR 137273-36-0/BI OR 13765-95-2/BI OR
               13765-96-3/BI OR 141-93-5/BI OR 141-97-9/BI OR 14309-92-3/BI
               OR 14414-90-5/BI OR 150-13-0/BI OR 15022-08-9/BI OR 156-43-4/BI
                OR 1864-93-3/BI OR 18707-60-3/BI OR 1943-8
L22       227422 SEA FILE=CAPLUS ABB=ON  PLU=ON  L21/PREP
L23           14 SEA FILE=CAPLUS ABB=ON  PLU=ON  L22 AND L13
L25            4 SEA FILE=CAPLUS ABB=ON  PLU=ON  L23 AND L19
L26            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L17 AND L19
L40           70 SEA FILE=CAPLUS ABB=ON  PLU=ON  SELVA M?/AU
L41          196 SEA FILE=CAPLUS ABB=ON  PLU=ON  TUNDO P?/AU
L51           86 SEA FILE=CAPLUS ABB=ON  PLU=ON  L18 AND L8
L52            6 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L10
L53         9721 SEA FILE=CAPLUS ABB=ON  PLU=ON  AMINES/CT (L) AROMATIC/OBI
L54            5 SEA FILE=CAPLUS ABB=ON  PLU=ON  L51 AND L53
L55            3 SEA FILE=CAPLUS ABB=ON  PLU=ON  L52 AND L54
L56            8 SEA FILE=CAPLUS ABB=ON  PLU=ON  L19 OR L25 OR L26 OR L54 OR
               L55
L57            4 SEA FILE=CAPLUS ABB=ON  PLU=ON  (L40 OR L41) AND L56

=> dup rem L39 L57
FILE 'CASREACT' ENTERED AT 16:00:38 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

FILE 'CAPLUS' ENTERED AT 16:00:38 ON 03 MAY 2006
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)
PROCESSING COMPLETED FOR L39
PROCESSING COMPLETED FOR L57
L61              9 DUP REM L39 L57 (2 DUPLICATES REMOVED)
                   ANSWERS '1-7' FROM FILE CASREACT
                   ANSWERS '8-9' FROM FILE CAPLUS

=> d ibib abs hit L61 1-7; d ibib abs hitind hitstr L61 8-9


L61  ANSWER 1 OF 9  CASREACT  COPYRIGHT 2006 ACS on STN DUPLICATE 1
ACCESSION NUMBER:          141:54061  CASREACT
TITLE:                     Process and catalysts for the synthesis of
                           mono-N-substituted functionalized anilines from
                           anilines and carbonate esters
```

INVENTOR(S): **Selva, Maurizio; Tundo, Pietro**
PATENT ASSIGNEE(S): Consorzio Interuniversitario Nazionale la Chimica per L'ambiente, Italy
SOURCE: Eur. Pat. Appl., 13 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

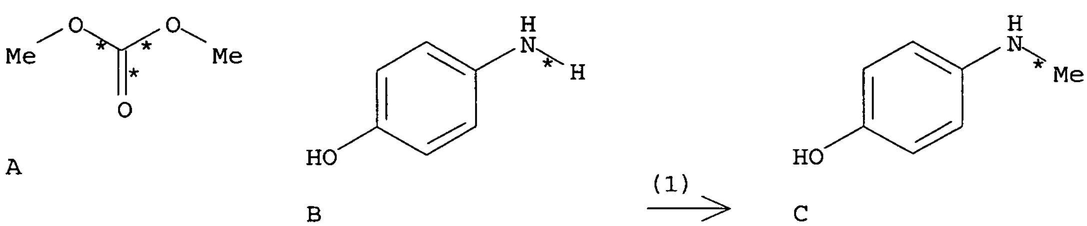
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1431274	A1	20040623	EP 2003-29005	20031216
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
US 2004127747	A1	20040701	US 2003-7~~34208~~	20031215
PRIORITY APPLN. INFO.:			IT 2002-PD325	20021218

OTHER SOURCE(S): MARPAT 141:54061

AB A process for direct and selective synthesis of mono-N-substituted functionalized anilines [e.g., 4-(methylamino)phenol] comprises the alkylation of anilines (e.g., 4-hydroxyaniline) with organic carbonates in the presence of faujasite-type zeolite catalysts that are chemical exchanged with alkali metals (e.g., sodium).

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 12 **A** + **B** ===> **C**



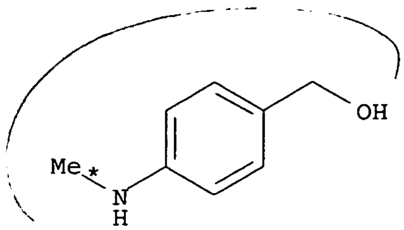
A

B

C
YIELD 91%

RX(1) RCT A **616-38-6**, B **123-30-8**
PRO C **150-75-4**
CAT 7440-23-5D Na
SOL 110-71-4 (CH2OMe)2
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 90 deg C
SUBSTAGE(3) 7 hours
NTE NaY-faujasite was used as catalyst

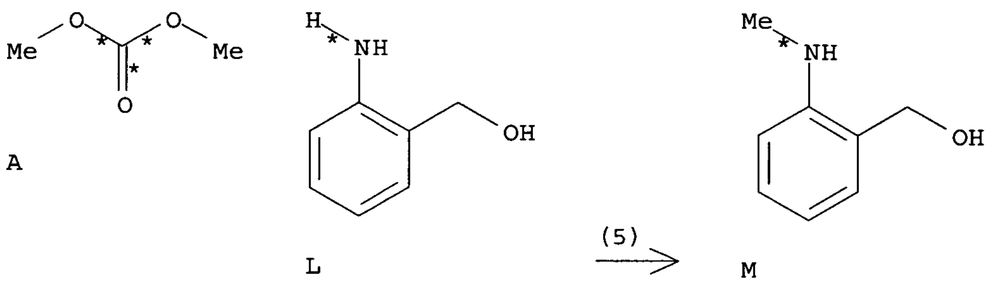
RX(2) OF 12 **A** + **F** ===> **G**



K
YIELD 77%

RX(4) RCT A **616-38-6**, J **623-04-1**
PRO K **181819-75-0**
CAT 7440-23-5D Na
SOL 616-38-6 Me2CO3
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 90 deg C
SUBSTAGE(3) 8 hours
NTE NaY-faujasite was used as catalyst

RX(5) OF 12 **A + L ===> M**



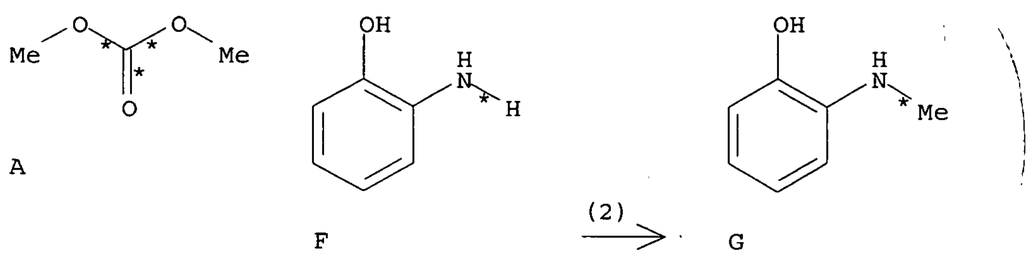
A

L

M
YIELD 92%

RX(5) RCT A **616-38-6**, L **5344-90-1**
PRO M **29055-08-1**
CAT 7440-23-5D Na
SOL 110-71-4 (CH2OMe)2
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 90 deg C
SUBSTAGE(3) 7 hours
NTE NaY-faujasite was used as catalyst

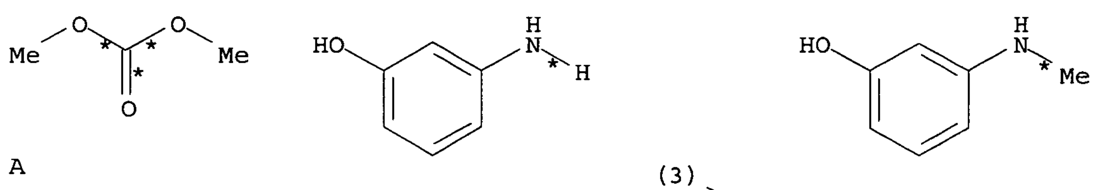
RX(6) OF 12 **A + N ===> O**



A F (2) G
YIELD 99%

```
RX(2)      RCT  A 616-38-6, F 95-55-6
           PRO  G 611-24-5
           CAT  7440-23-5D Na
           SOL  110-71-4 (CH2OMe)2
           CON  SUBSTAGE(1) room temperature
                SUBSTAGE(2) room temperature -> 90 deg C
                SUBSTAGE(3) 3 hours
           NTE  NaY-faujasite was used as catalyst
```

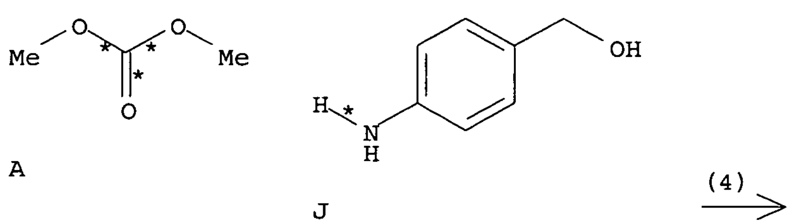
RX(3) OF 12 **A** + **H** ===> **I**



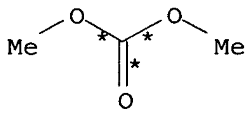
A H (3) I
YIELD 92%

```
RX(3)      RCT  A 616-38-6, H 591-27-5
           PRO  I 14703-69-6
           CAT  7440-23-5D Na
           SOL  616-38-6 Me2CO3
           CON  SUBSTAGE(1) room temperature
                SUBSTAGE(2) room temperature -> 90 deg C
                SUBSTAGE(3) 7 hours
           NTE  NaY-faujasite was used as catalyst
```

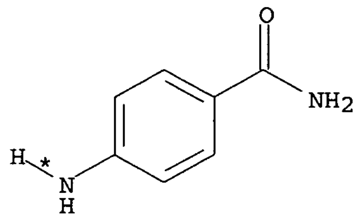
RX(4) OF 12 **A** + **J** ===> **K**



A J (4)

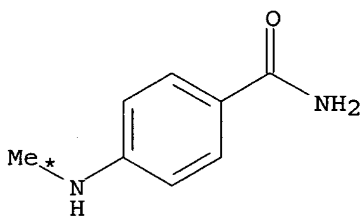


A



N

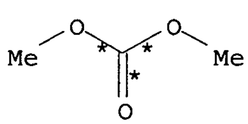
(6) ⟶



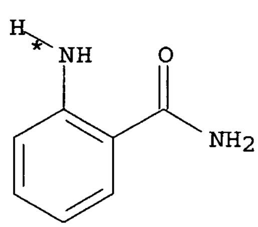
O
YIELD 86%

```
RX(6)       RCT  A 616-38-6, N 2835-68-9
            PRO  O 38359-26-1
            CAT  7440-23-5D Na
            SOL  110-71-4 (CH2OMe)2
            CON  SUBSTAGE(1) room temperature
                 SUBSTAGE(2) room temperature -> 90 deg C
                 SUBSTAGE(3) 7 hours
            NTE  NaY-faujasite was used as catalyst
```

RX(7) OF 12 **A + P ===> Q**

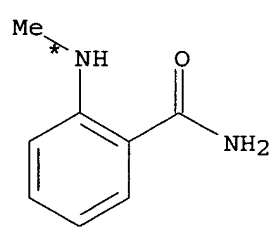


A



P

(7) ⟶

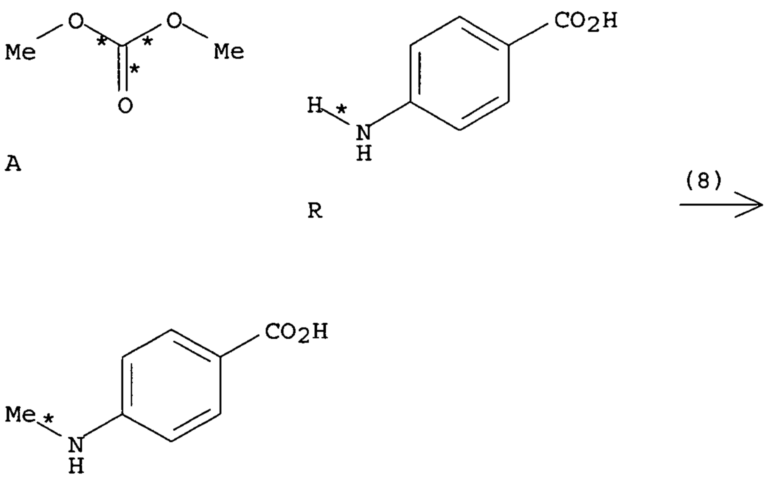


Q
YIELD 91%

```
RX(7)       RCT  A 616-38-6, P 88-68-6
            PRO  Q 7505-81-9
            CAT  7440-23-5D Na
            SOL  110-71-4 (CH2OMe)2
```

CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 90 deg C
SUBSTAGE(3) 22 hours
NTE NaY-faujasite was used as catalyst

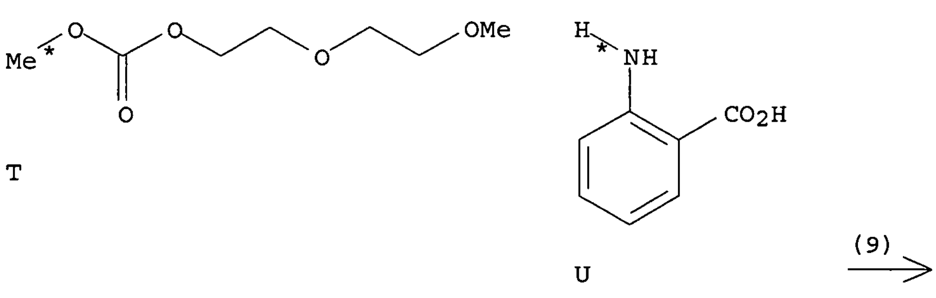
RX(8) OF 12 **A + R ===> S**

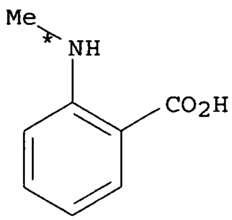


S
YIELD 74%

RX(8) RCT A **616-38-6**, R **150-13-0**
PRO S **10541-83-0**
CAT 7440-23-5D Na
SOL 110-71-4 (CH2OMe)2
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 130 deg C
SUBSTAGE(3) 9 hours
NTE NaY-faujasite was used as catalyst

RX(9) OF 12 **T + U ===> V**

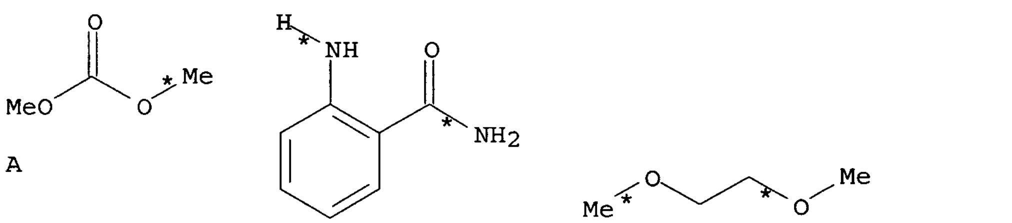




V
YIELD 93%

```
RX(9)      RCT  T 141814-27-9, U 118-92-3
           PRO  V 119-68-6
           CAT  7440-23-5D Na
           SOL  112-49-2 Triglyme
           CON  SUBSTAGE(1) room temperature
                SUBSTAGE(2) room temperature -> 150 deg C
                SUBSTAGE(3) 12 hours
           NTE  NaY-faujasite was used as catalyst, thermal
```

RX(10) OF 12   **A** + **P** + E ===> **X**

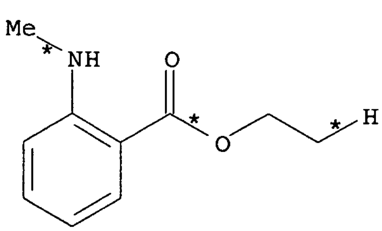


A

P

E

(10)

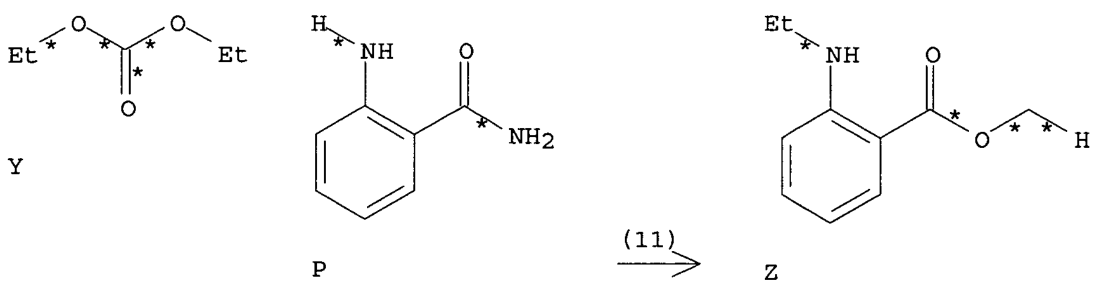


X
YIELD 65%

```
RX(10)     RCT  A 616-38-6, P 88-68-6, E 110-71-4
           PRO  X 35472-56-1
           CAT  7440-23-5D Na
           SOL  110-71-4 (CH2OMe)2
           CON  SUBSTAGE(1) room temperature
```

SUBSTAGE(2) room temperature -> 150 deg C
SUBSTAGE(3) 8 hours
NTE NaY-faujasite was used as catalyst, thermal

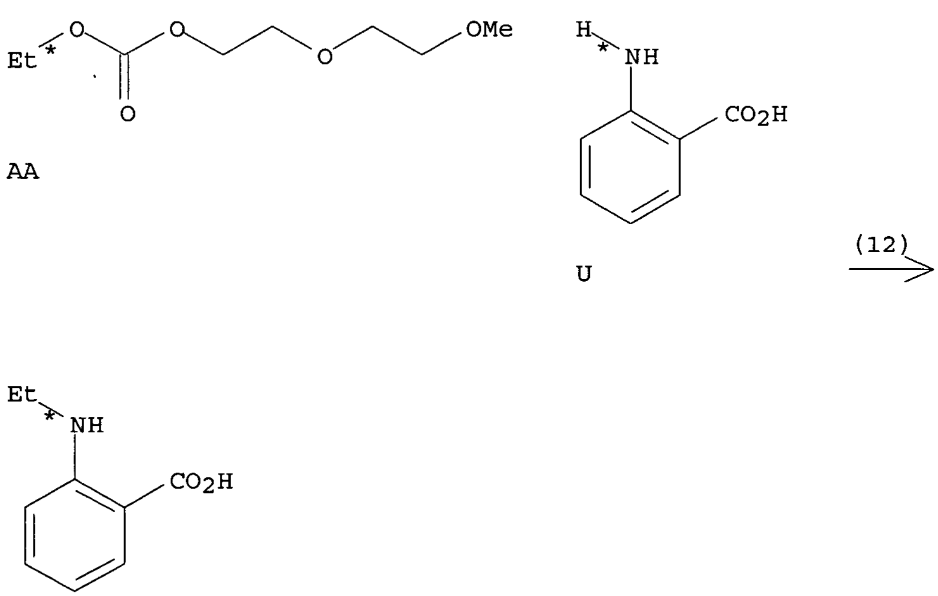
RX(11) OF 12 **Y** + **P** ===> **Z**



Y P Z YIELD 23%

RX(11) RCT Y **105-58-8**, P **88-68-6**
PRO Z **17318-49-9**
CAT 7440-23-5D Na
SOL 110-71-4 (CH2OMe)2
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 150 deg C
SUBSTAGE(3) 8 hours
NTE NaY-faujasite was used as catalyst, thermal

RX(12) OF 12 **AA** + **U** ===> **AB**



AA U AB

RX(12) RCT AA **214470-03-8**, U **118-92-3**

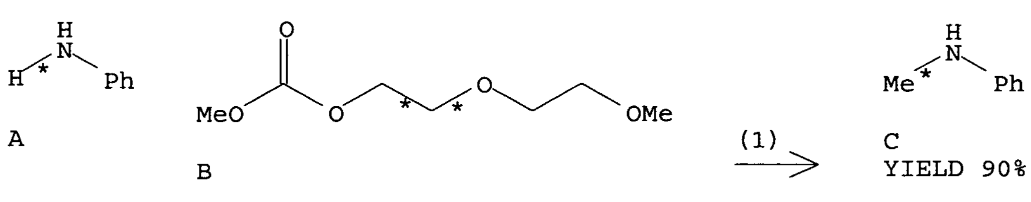
PRO AB **89-50-9**
CAT 7440-23-5D Na
SOL 112-49-2 Triglyme
CON SUBSTAGE(1) room temperature
SUBSTAGE(2) room temperature -> 150 deg C
SUBSTAGE(3) 18 hours
NTE NaY-faujasite was used as catalyst, thermal
IN **Selva, Maurizio; Tundo, Pietro**

L61 ANSWER 2 OF 9 CASREACT COPYRIGHT 2006 ACS on STN DUPLICATE 2
ACCESSION NUMBER: 134:222468 CASREACT
TITLE: Reaction of Primary Aromatic Amines with Alkyl Carbonates over NaY Faujasite: A Convenient and Selective Access to Mono-N-alkyl Anilines
AUTHOR(S): **Selva, Maurizio; Tundo, Pietro**; Perosa, Alvise
CORPORATE SOURCE: Dipartimento di Scienze Ambientali, Universita Ca' Foscari, Venice, 2137-30123, Italy
SOURCE: Journal of Organic Chemistry (2001), 66(3), 677-680
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB At atmospheric pressure and at 130-160 °C, primary aromatic amines (p-XC6H4NH2, X = H, Cl, NO2) are mono-N-alkylated in a single step, with sym. and asym. dialkyl carbonates [ROCOOR', R = Me, R' = MeO(CH2)2O(CH2)2; R = R' = Et; R = R' = benzyl; R = R' = allyl; R = Et, R' = MeO(CH2)2O(CH2)2], in the presence of a com. available NaY faujasite. No solvents are required. Mono-N-alkyl anilines are obtained with a very high selectivity (90-97%), in good to excellent yields (68-94%), on a preparative scale. In the presence of triglyme as a solvent, the mono-N-alkyl selectivity is independent of concentration and polarity factors. The reaction probably takes place within the polar zeolite cavities, and through the combined effect of the dual acid-base properties of the catalyst.

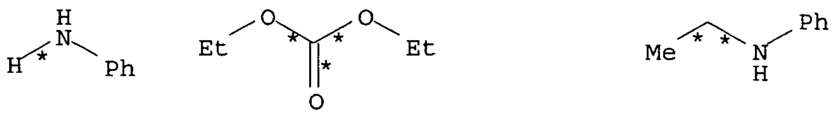
REFERENCE COUNT: 48 THERE ARE 48 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 12 **A** + **B** ===> **C**



RX(1) RCT A **62-53-3**, B **141814-27-9**
PRO C **100-61-8**
NTE NaY faujasite catalyst used; no solvent; thermal; regioselective; green chem.-waste redn.

RX(2) OF 12 **A** + 2 **D** ===> **E**



A

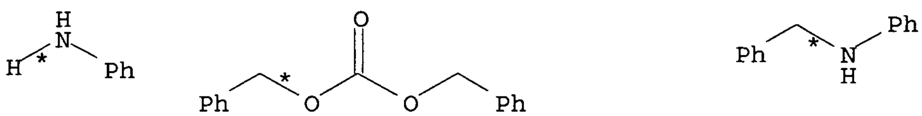
2 D

(2)

E
YIELD 84%

RX(2)   RCT A **62-53-3**, D **105-58-8**
          PRO E **103-69-5**
          NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

RX(3) OF 12   **A + F ===> G**



A

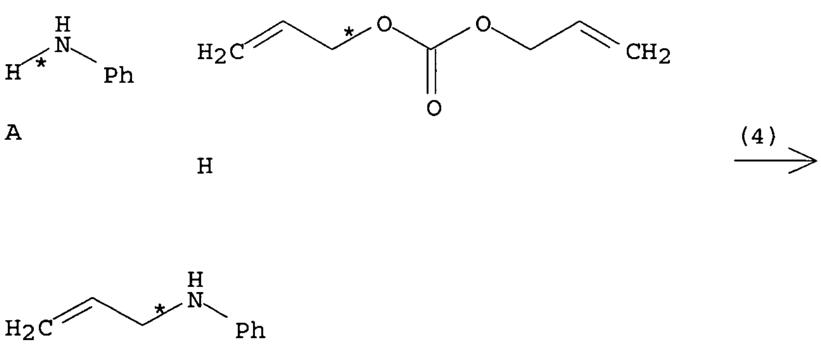
F

(3)

G
YIELD 91%

RX(3)   RCT A **62-53-3**, F **3459-92-5**
          PRO G **103-32-2**
          NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

RX(4) OF 12   **A + H ===> I**



A

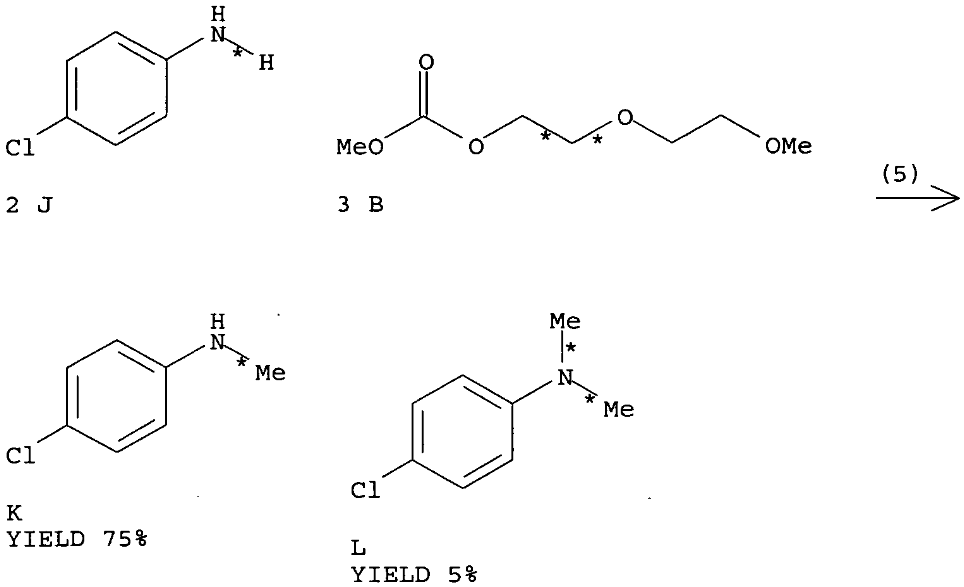
H

(4)

I
YIELD 94%

RX(4)   RCT A **62-53-3**, H **15022-08-9**
          PRO I **589-09-3**
          NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

RX(5) OF 12 2 **J** + 3 **B** ===> **K** + L



2 J

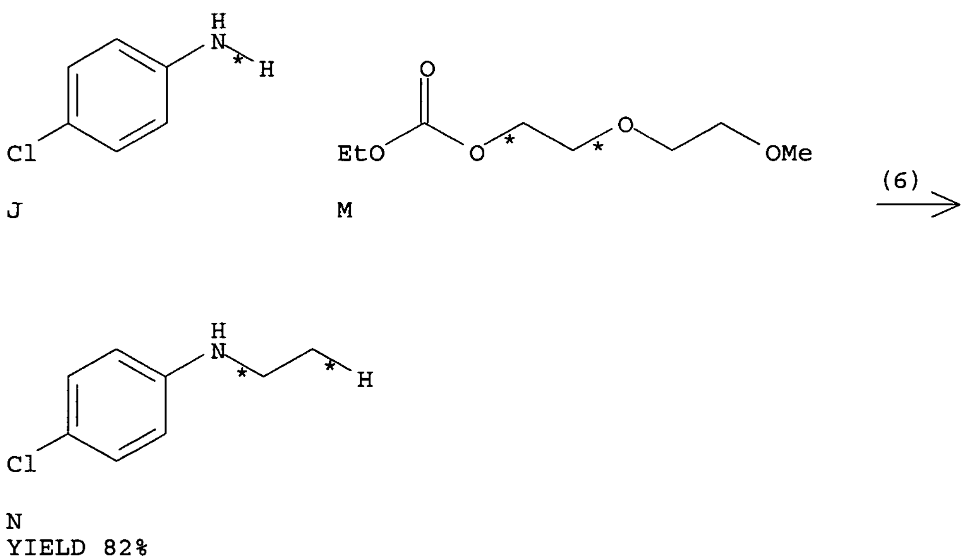
3 B

K
YIELD 75%

L
YIELD 5%

RX(5) RCT J **106-47-8**, B **141814-27-9**
PRO K **932-96-7**, L 698-69-1
NTE NaY faujasite catalyst used; no solvent; thermal; regioselective; green chem.-waste redn.

RX(6) OF 12 **J** + **M** ===> **N**



J

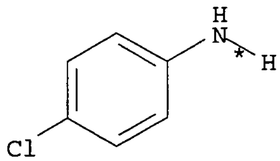
M

N
YIELD 82%

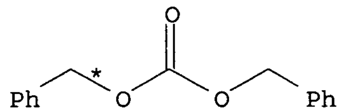
RX(6) RCT J **106-47-8**, M **214470-03-8**
PRO N **13519-75-0**
NTE NaY faujasite catalyst used; no solvent; thermal;

regioselective; green chem.-waste redn.

RX(7) OF 12 **J** + **F** ===> **O**

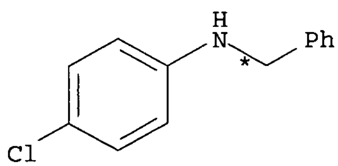


J



F

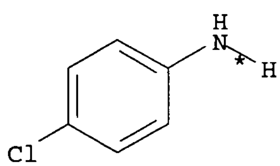
(7) →



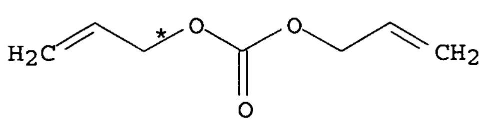
O
YIELD 70%

RX(7) RCT J **106-47-8**, F **3459-92-5**
PRO O **2948-37-0**
NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

RX(8) OF 12 2 **J** + 3 **H** ===> **P** + Q

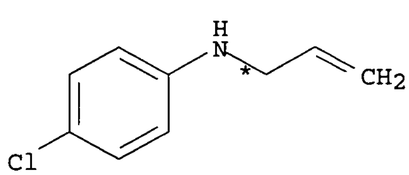


2 J

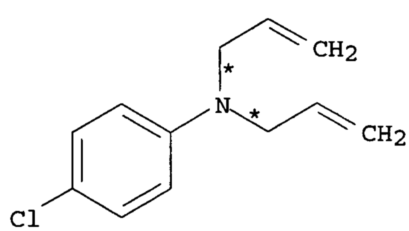


3 H

(8) →



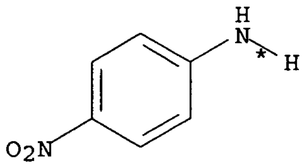
P
YIELD 82%



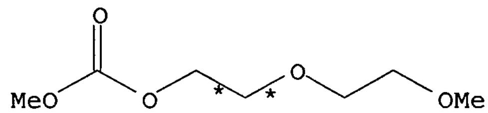
Q
YIELD 5%

RX(8) RCT J **106-47-8**, H **15022-08-9**
PRO P **13519-80-7**, Q 30438-94-9
NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

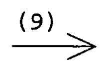
RX(9) OF 12 2 **R** + 3 **B** ===> **S** + T

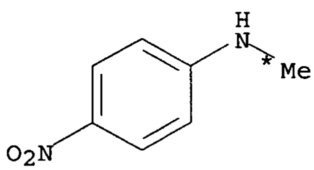


2 R

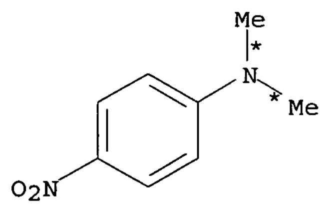


3 B





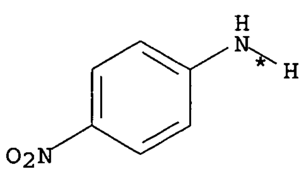
S
YIELD 80%



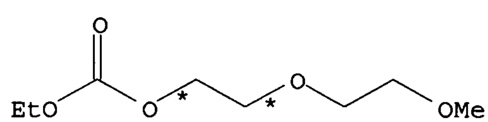
T
YIELD 7%

RX(9) RCT R **100-01-6**, B **141814-27-9**
PRO S **100-15-2**, T 100-23-2
NTE NaY faujasite catalyst used; no solvent; thermal; regioselective; green chem.-waste redn.

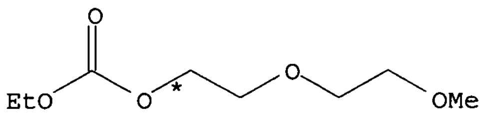
RX(10) OF 12 2 **R** + 2 **M** ===> **U** + V



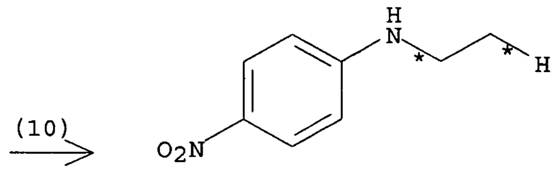
2 R



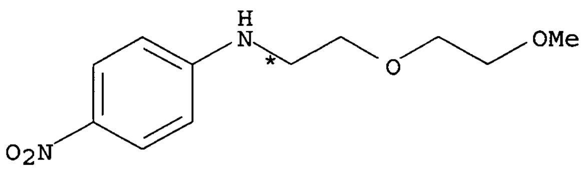
M



M



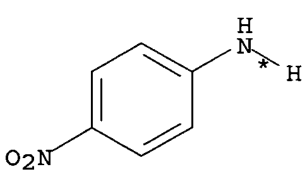
U
YIELD 68%



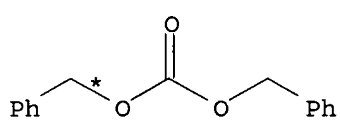
V
YIELD 10%

RX(10)   RCT R **100-01-6**, M **214470-03-8**
           PRO U **3665-80-3**, V 329767-78-4
           NTE NaY faujasite catalyst used; no solvent; thermal;
                regioselective; green chem.-waste   redn.

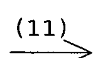
RX(11) OF 12   **R** + **F** ===> **W**

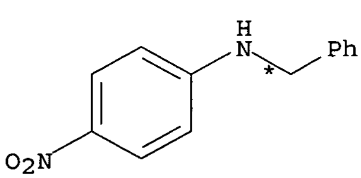


R



F



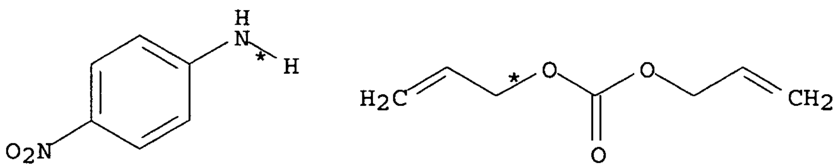


W
YIELD 78%

RX(11)   RCT R **100-01-6**, F **3459-92-5**
           PRO W **14309-92-3**

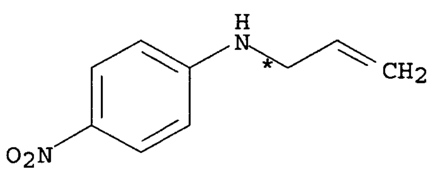
NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.

RX(12) OF 12 **R** + **H** ===> **X**



R H

(12) →



X
YIELD 78%

RX(12) RCT R **100-01-6**, H **15022-08-9**
PRO X **4138-40-3**
NTE NaY faujasite catalyst used; no solvent; thermal; green chem.-waste redn.
AU **Selva, Maurizio**; **Tundo, Pietro**; Perosa, Alvise

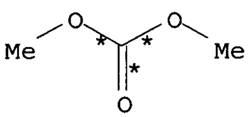
L61 ANSWER 3 OF 9 CASREACT

ACCESSION NUMBER: 142:463238 CASREACT
TITLE: Mono-N-methylation of Functionalized Anilines with Alkyl Methyl Carbonates over NaY Faujasites. 4. Kinetics and Selectivity
AUTHOR(S): **Selva, Maurizio**; **Tundo, Pietro**; Foccardi, Tommaso
CORPORATE SOURCE: Dipartimento di Scienze Ambientali, Universita Ca' Foscari, Venice, 30123, Italy
SOURCE: Journal of Organic Chemistry (2005), 70(7), 2476-2485
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
AB In the presence of NaY faujasite as the catalyst, the reaction of bifunctional anilines (1-4: XC6H4NH2; X = OH, CO2H, CH2OH, and CONH2) with Me alkyl carbonates [MeOCO2R': R' = Me or MeO(CH2)2O(CH2)2] proceeds with a very high mono-N-Me selectivity (XC6H4NHMe up to 99%), and chemoselectivity as well, with other nucleophilic functions (OH, CO2H, CH2OH, CONH2) fully preserved from alkylation and/or transesterification reactions. Aromatic substituents, however, modify the relative reactivity of amines 1-4: good evidence suggests that, not only steric and electronic effects, but, importantly, direct acid-base interactions between

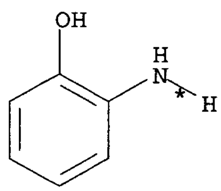
substituents and the catalyst are involved. Weakly acidic groups (OH, CH2OH, CONH2, pKa ≥ 10) may help the reaction, while aminobenzoic acids (pKa of 4-5) are the least reactive substrates. The solvent polarity also affects the reaction, which is faster in xylene than in the more polar diglyme. The mono-N-Me selectivity is explained by the adsorption pattern of reagents within the zeolite pores: a BAl2 displacement of the amine on Me alkyl carbonate should occur aided by the geometric features of the NaY supercavities. Different factors account for the reaction chemoselectivity. The polarizability of the two nucleophilic terms (NH2 and X groups) of anilines is relevant, although adsorption and confinement phenomena of reagents promoted by the zeolite should also be considered.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 21 3 **A** + 2 **B** ===> **C** + D

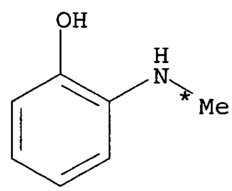


3 A

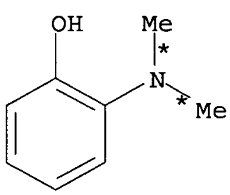


2 B

(1) →



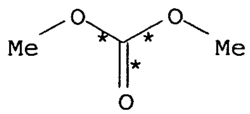
C



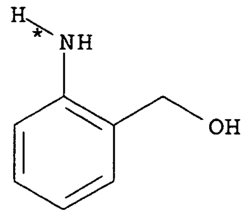
D

RX(1) RCT A **616-38-6**, B **95-55-6**
PRO C **611-24-5**, D 3743-22-4
SOL 616-38-6 Me2CO3
CON 150 minutes, 90 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(2) OF 21 3 **A** + 2 **E** ===> **F** + G

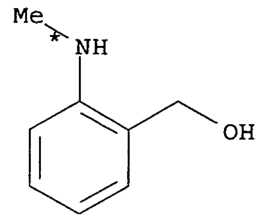


3 A

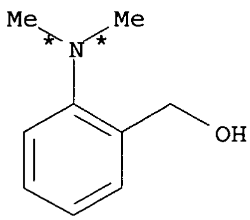


2 E

(2) →



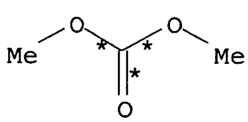
F



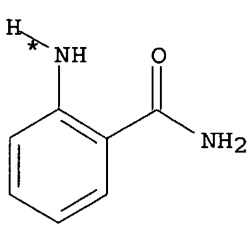
G

RX(2) RCT A **616-38-6**, E **5344-90-1**
PRO F **29055-08-1**, G 4707-56-6
SOL 616-38-6 Me2CO3
CON 405 minutes, 90 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(3) OF 21 3 **A** + 2 **H** ===> **I** + J

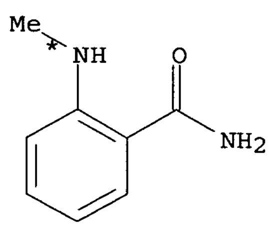


3 A

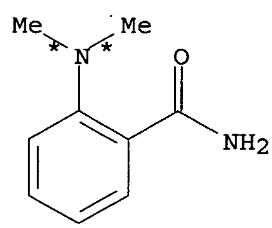


2 H

(3) →



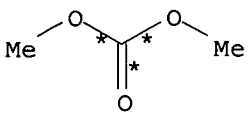
I



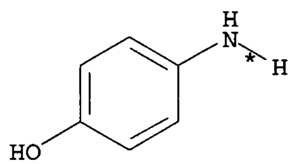
J

RX(3) RCT A **616-38-6**, H **88-68-6**
PRO I **7505-81-9**, J 56042-77-4
SOL 616-38-6 Me2CO3
CON 540 minutes, 90 deg C
NTE regioselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(4) OF 21 3 **A** + 2 **K** ===> **L** + M

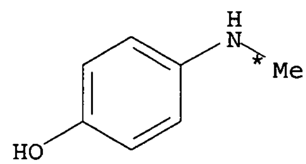


3 A

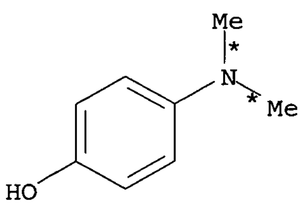


2 K

(4)



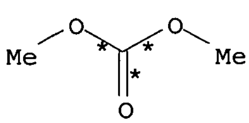
L



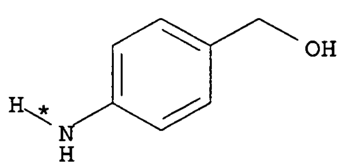
M

RX(4) RCT A **616-38-6**, K **123-30-8**
PRO L **150-75-4**, M 619-60-3
SOL 616-38-6 Me2CO3, 110-71-4 (CH2OMe)2
CON 315 minutes, 84 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(5) OF 21 3 **A** + 2 **O** ===> **P** + Q

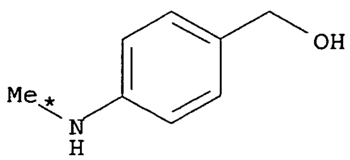


3 A

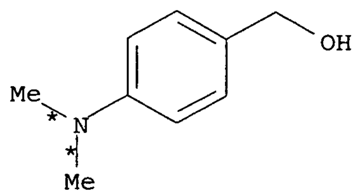


2 O

(5)



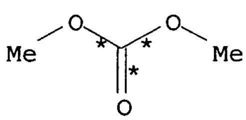
P



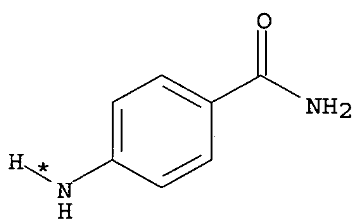
Q

RX(5) RCT A **616-38-6**, O **623-04-1**
PRO P **181819-75-0**, Q 1703-46-4
SOL 616-38-6 Me2CO3, 110-71-4 (CH2OMe)2
CON 570 minutes, 84 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

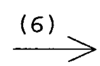
RX(6) OF 21 3 **A** + 2 **R** ===> **S** + T.

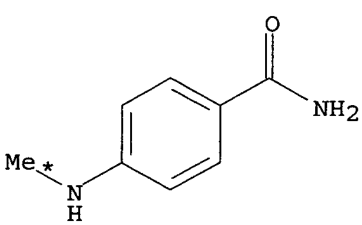


3 A

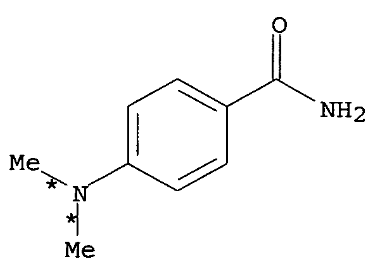


2 R





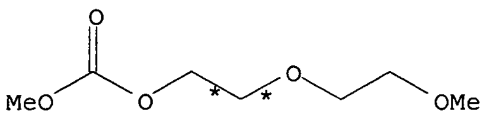
S



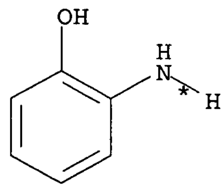
T

RX(6) RCT A **616-38-6**, R **2835-68-9**
PRO S **38359-26-1**, T 6083-47-2
SOL 616-38-6 Me2CO3, 110-71-4 (CH2OMe)2
CON 570 minutes, 84 deg C
NTE regioselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(7) OF 21 3 **U** + 2 **B** ===> **C** + D

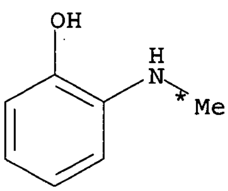


3 U

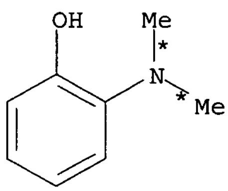


2 B

$\xrightarrow{(7)}$



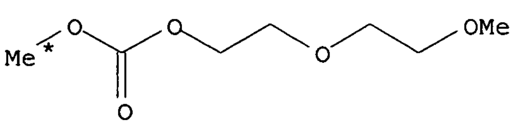
C



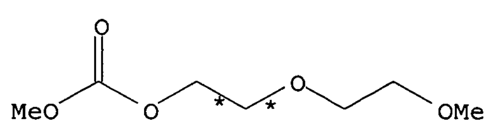
D

RX(7) RCT U **141814-27-9**, B **95-55-6**
PRO C **611-24-5**, D 3743-22-4
SOL 1330-20-7 Xylene
CON 10 minutes, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

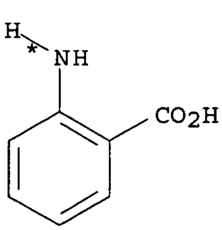
RX(8) OF 21 3 **U** + 2 **W** ===> **X** + Y



U

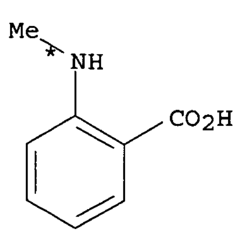


2 U

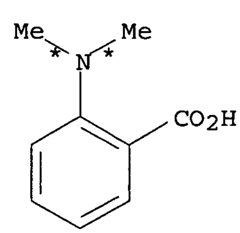


2 W

$\xrightarrow{(8)}$



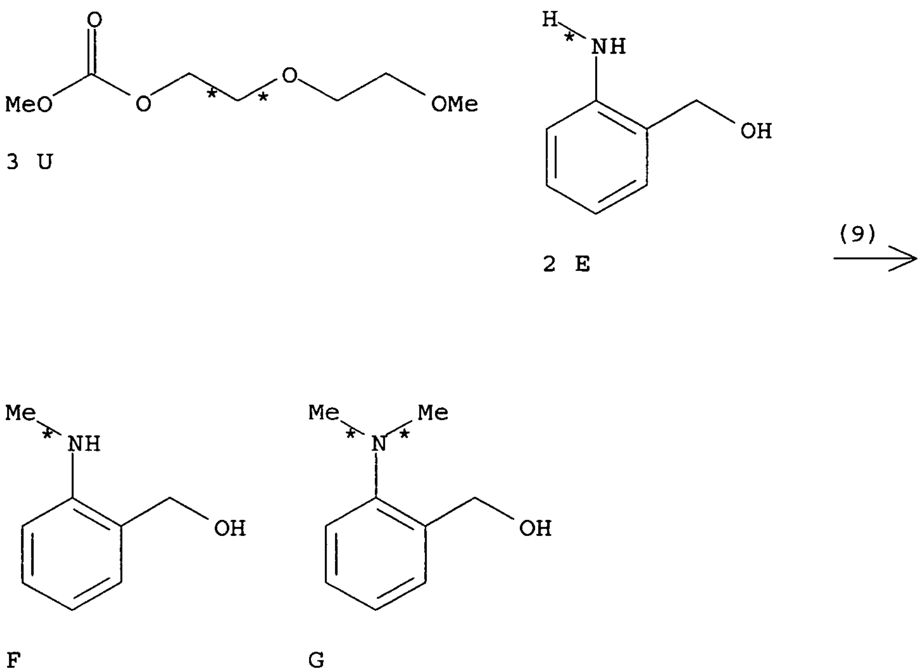
X



Y

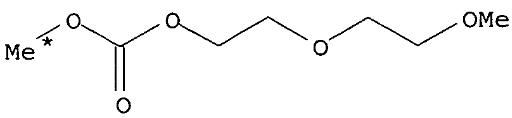
RX(8) RCT U **141814-27-9**, W **118-92-3**
PRO X **119-68-6**, Y 610-16-2
SOL 1330-20-7 Xylene
CON 540 minutes, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(9) OF 21 3 **U** + 2 **E** ===> **F** + G

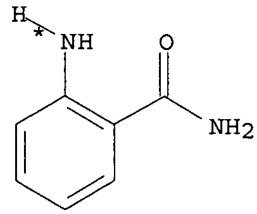


RX(9) RCT U **141814-27-9**, E **5344-90-1**
PRO F **29055-08-1**, G 4707-56-6
SOL 1330-20-7 Xylene
CON 80 minutes, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(10) OF 21 3 **U** + 2 **H** ===> **I** + J

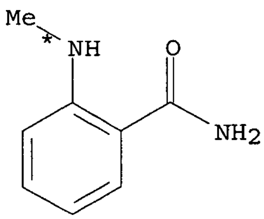


3 U

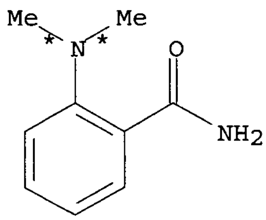


2 H

(10) →



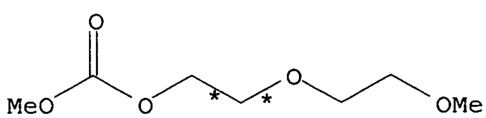
I



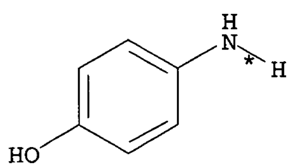
J

RX(10) RCT U **141814-27-9**, H **88-68-6**
PRO I **7505-81-9**, J 56042-77-4
SOL 1330-20-7 Xylene
CON 80 minutes, 135 deg C
NTE regioselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

RX(11) OF 21 3 **U** + 2 **K** ===> **L** + M

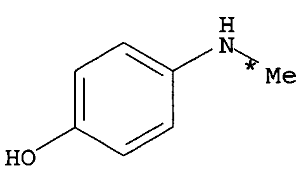


3 U

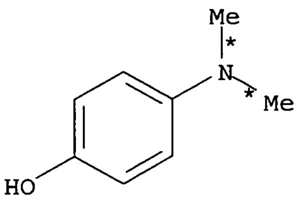


2 K

(11) →



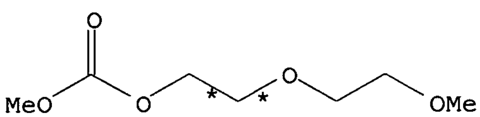
L



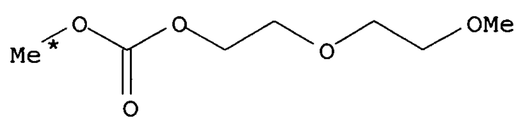
M

RX(11) RCT U **141814-27-9**, K **123-30-8**
PRO L **150-75-4**, M 619-60-3
SOL 111-96-6 (MeOCH2CH2)2O
CON 50 minutes, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

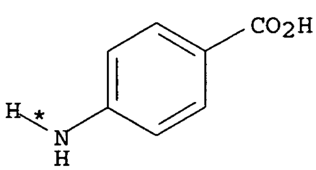
RX(12) OF 21 3 **U** + 2 **AA** ===> **AB** + AC



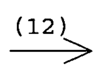
2 U

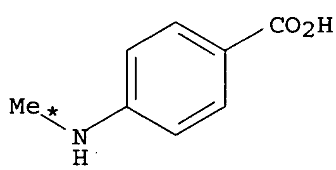


U

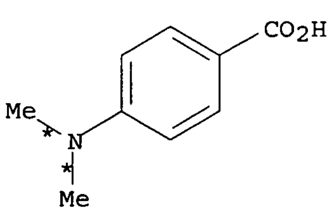


2 AA





AB

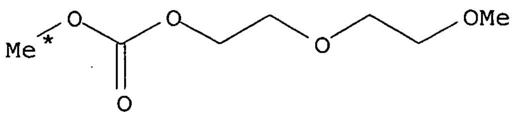


AC

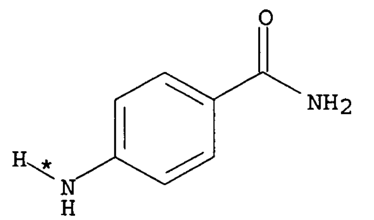
RX(12) RCT U **141814-27-9**, AA **150-13-0**
PRO AB **10541-83-0**, AC 619-84-1
SOL 111-96-6 (MeOCH2CH2)2O
CON 600 minutes, 135 deg C

NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

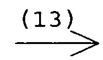
RX(13) OF 21 3 **U** + 2 **R** ===> **S** + T

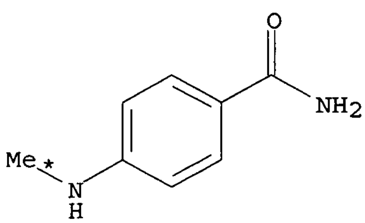


3 U

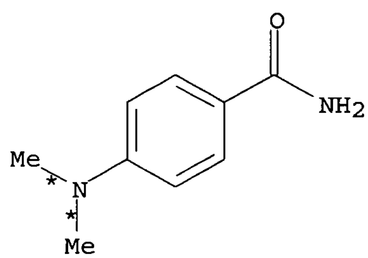


2 R





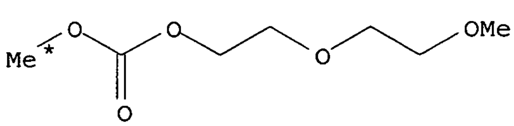
S



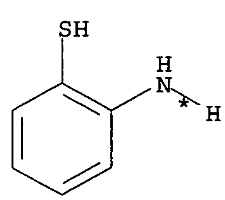
T

RX(13) RCT U **141814-27-9**, R **2835-68-9**
PRO S **38359-26-1**, T 6083-47-2
SOL 111-96-6 (MeOCH2CH2)2O
CON 50 minutes, 135 deg C
NTE regioselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation, alternate preparation described

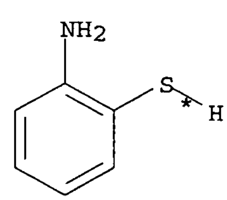
RX(14) OF 21 7 **U** + 4 **AD** ===> **AE** + AF + AG + AH



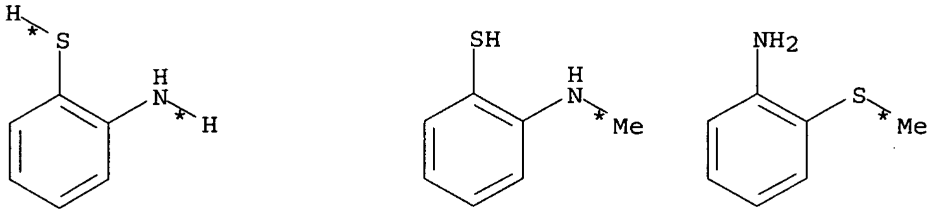
7 U



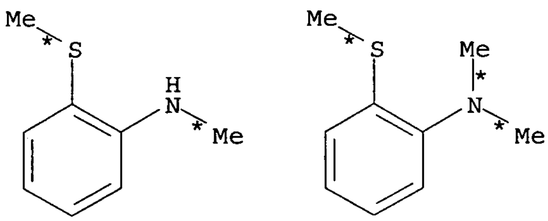
AD



AD



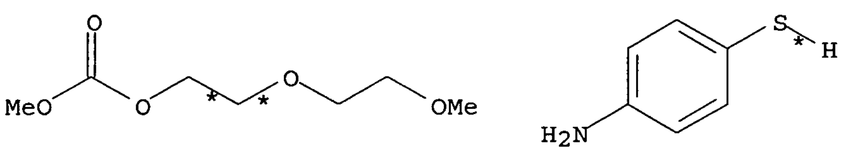
2 AD $\xrightarrow{(14)}$ AE AF



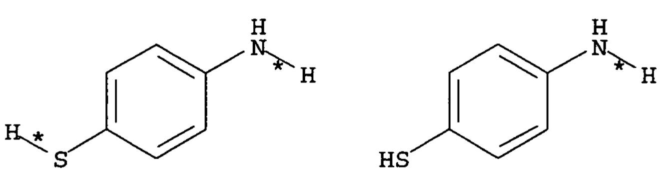
AG AH

RX(14) RCT U **141814-27-9**, AD **137-07-5**
PRO AE **21749-63-3**, AF 2987-53-3, AG 13372-62-8, AH 2388-50-3
SOL 1330-20-7 Xylene
CON 8 hours, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used

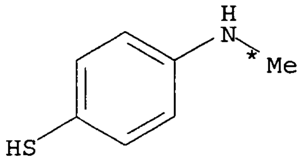
RX(15) OF 21 6 **U** + 4 **AI** ===> **AJ** + AK +
AL + AM



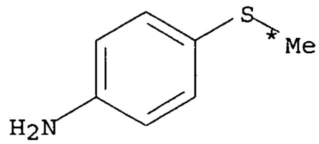
6 U AI



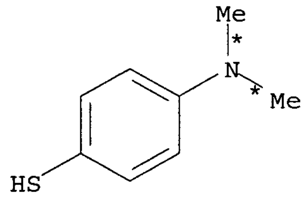
AI 2 AI $\xrightarrow{(15)}$



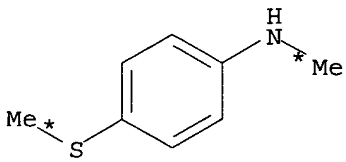
AJ



AK



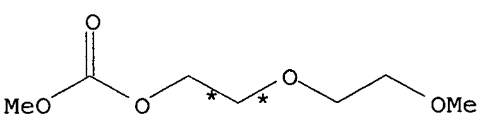
AL



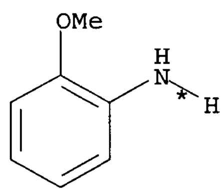
AM

RX(15) RCT U **141814-27-9**, AI **1193-02-8**
PRO AJ **4946-21-8**, AK 104-96-1, AL 4946-22-9, AM 58259-33-9
SOL 1330-20-7 Xylene
CON 4 hours, 135 deg C
NTE chemoselective, solid-supported catalyst, kinetic study, NaY Faujasite catalyst used

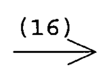
RX(16) OF 21 3 **U** + 2 **AN** ===> **AO** + AP

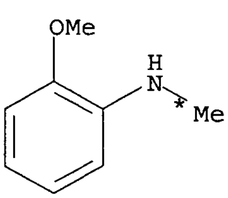


3 U

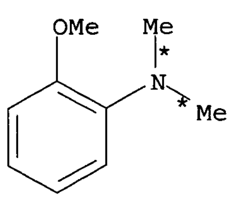


2 AN





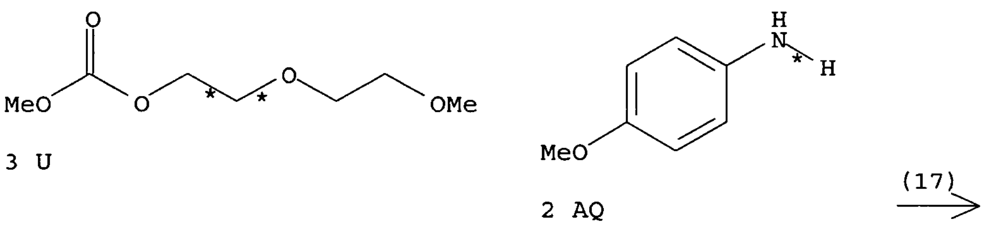
AO



AP

RX(16) RCT U **141814-27-9**, AN **90-04-0**
PRO AO **10541-78-3**, AP 700-75-4
SOL 1330-20-7 Xylene
CON 1320 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

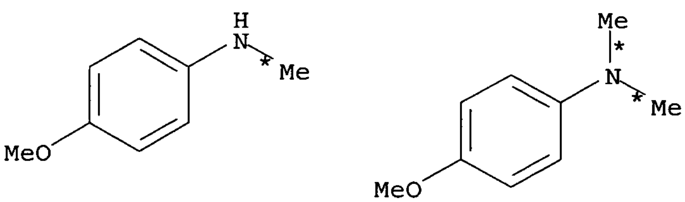
RX(17) OF 21 3 **U** + 2 **AQ** ===> **AR** + AS



3 U

2 AQ

(17)

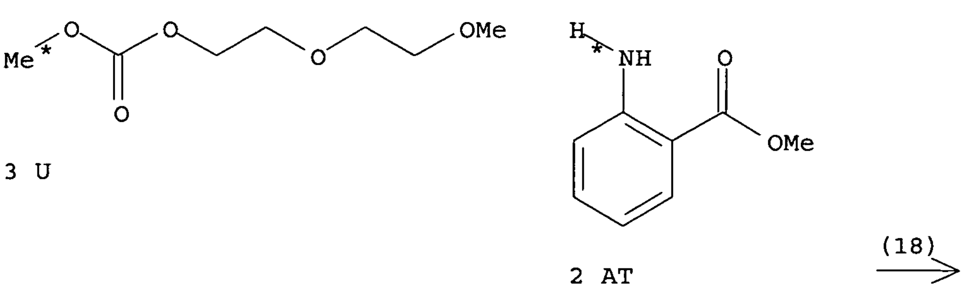


AR

AS

RX(17) RCT U **141814-27-9**, AQ **104-94-9**
PRO AR **5961-59-1**, AS 701-56-4
SOL 1330-20-7 Xylene
CON 210 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

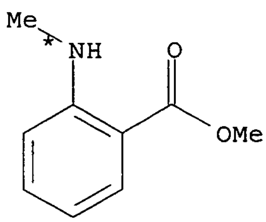
RX(18) OF 21 3 **U** + 2 **AT** ===> **AU** + AV



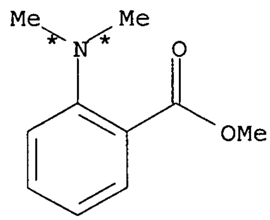
3 U

2 AT

(18)



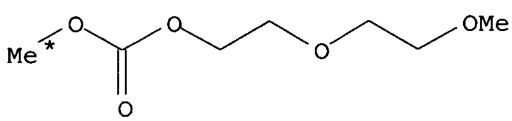
AU



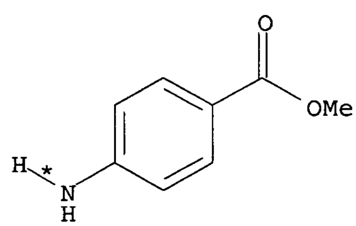
AV

RX(18) RCT U **141814-27-9**, AT **134-20-3**
PRO AU **85-91-6**, AV 10072-05-6
SOL 1330-20-7 Xylene
CON 660 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

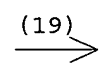
RX(19) OF 21 3 **U** + 2 **AW** ===> **AX** + AY

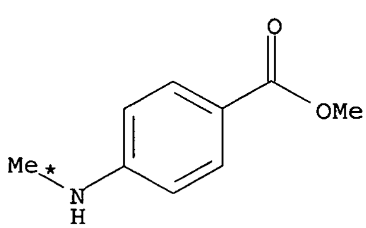


3 U

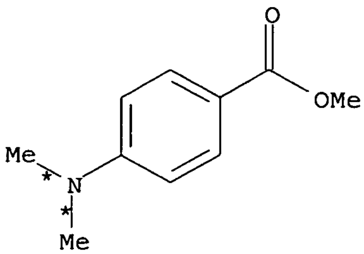


2 AW





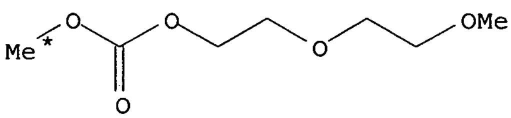
AX



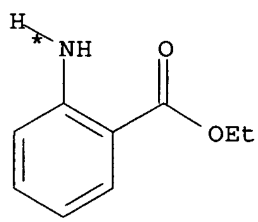
AY

RX(19) RCT U **141814-27-9**, AW **619-45-4**
PRO AX **18358-63-9**, AY 1202-25-1
SOL 1330-20-7 Xylene
CON 270 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

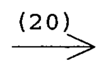
RX(20) OF 21 3 **U** + 2 **AZ** ===> **BA** + BB

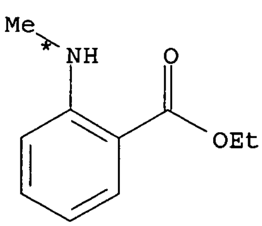


3 U

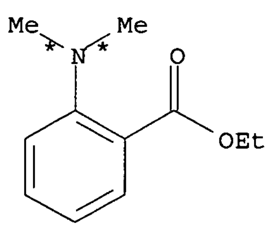


2 AZ





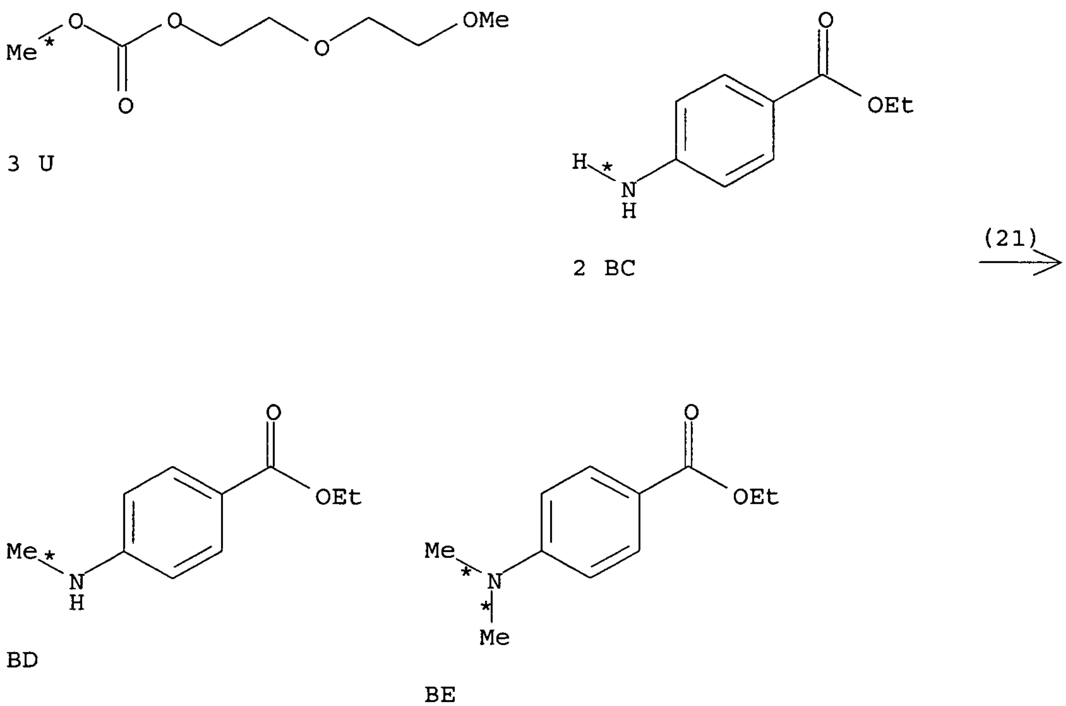
BA



BB

RX(20) RCT U **141814-27-9**, AZ **87-25-2**
PRO BA **35472-56-1**, BB 55426-74-9
SOL 1330-20-7 Xylene
CON 4680 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation

RX(21) OF 21 3 **U** + 2 **BC** ===> **BD** + BE



RX(21) RCT U **141814-27-9**, BC **94-09-7**
PRO BD **10541-82-9**, BE 10287-53-3
SOL 1330-20-7 Xylene
CON 540 minutes, 135 deg C
NTE solid-supported catalyst, kinetic study, NaY Faujasite catalyst used, selective to mono-N-methylation
AU **Selva, Maurizio**; **Tundo, Pietro**; Foccardi, Tommaso

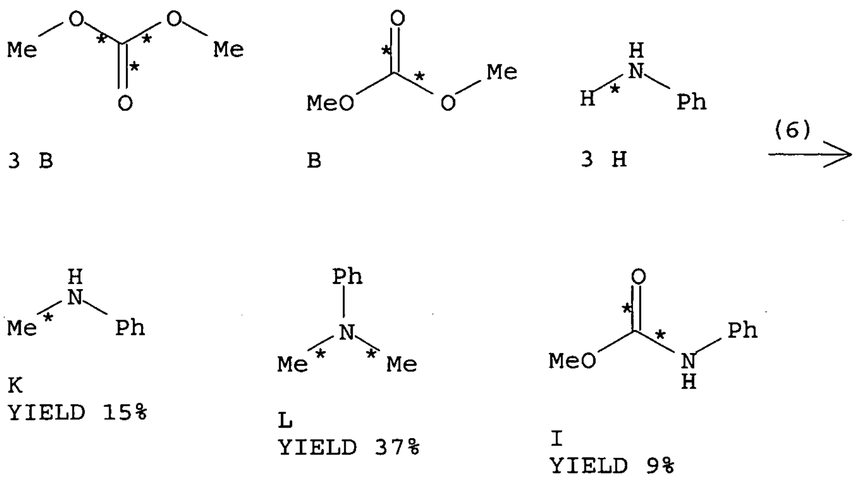
L61 ANSWER 4 OF 9 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 142:335922 CASREACT
TITLE: Dimethyl Carbonate as an Ambident Electrophile
AUTHOR(S): **Tundo, Pietro**; Rossi, Laura; Loris, Alessandro
CORPORATE SOURCE: Dipartimento di Scienze Ambientali, Universita Ca' Foscari, Venice, Italy
SOURCE: Journal of Organic Chemistry (2005), 70(6), 2219-2224
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The features of various anions having different soft/hard character (aliphatic and aromatic amines, alkoxides, phenoxides, thiolates) are compared with regard to nucleophilic substitutions on di-Me carbonate (DMC), using different reaction conditions. Results are well in agreement with the Hard-Soft Acid-Base (HSAB) theory. Accordingly, the high selectivity of monomethylation of CH2 acidic compds. and primary aromatic amines with DMC can be explained by two different subsequent reactions, which are due to the double electrophilic character of DMC. The first step consists of a hard-hard reaction and selectively produces a soft anion, which, in the

second phase, selectively transforms into the final monomethylated product, via a soft-soft nucleophilic displacement (yield >99% at complete conversion, using DMC as solvent).

REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(6) OF 17 4 **B** + 3 **H** ===> **K** + L + I



RX(6) RCT B **616-38-6**, H **62-53-3**
PRO K **100-61-8**, L 121-69-7, I 2603-10-3
CON 24 hours, 200 deg C
NTE chemoselective, high pressure
AU **Tundo, Pietro**; Rossi, Laura; Loris, Alessandro

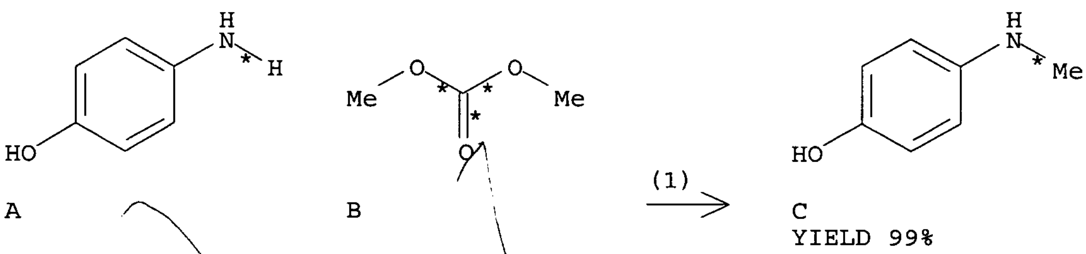
L61 ANSWER 5 OF 9 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 139:323290 CASREACT
TITLE: Reaction of Functionalized Anilines with Dimethyl Carbonate over NaY Faujasite. 3. Chemoselectivity toward Mono-N-methylation
AUTHOR(S): **Selva, Maurizio**; **Tundo, Pietro**; Perosa, Alvise
CORPORATE SOURCE: Dipartimento di Scienze Ambientali, Universita Ca' Foscari, Venice, 30123, Italy
SOURCE: Journal of Organic Chemistry (2003), 68(19), 7374-7378 CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB In the presence of NaY faujasite, di-Me carbonate (MeOCO2Me, DMC) is a highly chemoselective methylating agent of functionalized anilines such as aminophenols, aminobenzyl alcs., aminobenzoic acids, and aminobenzamides. The reaction proceeds with the exclusive formation of N-methylanilines without any concurrent O-methylation or N-/O-methoxy carbonylation side processes. Particularly, only mono-N-Me derivs. [XC6H4(1:1)NHMe, X = o-, m-, and p-OH; o- and p-CH2OH; o- and p-CO2H; o- and p-CONH2] are obtained with selectivity up to 99% and isolated yields of 74-99%. DMC, which usually promotes methylations only at T > 120 °C, is activated by (1:1)the zeolite catalyst and it reacts with all aniline compds. at 90

°C. Aminobenzoic acids require a higher reaction temperature
(≥130 °C).

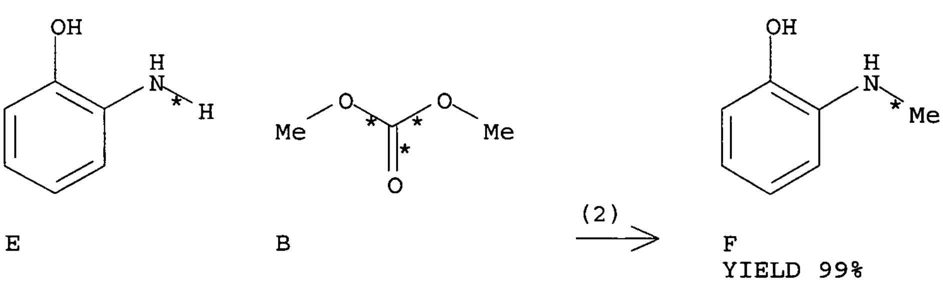
REFERENCE COUNT: 65 THERE ARE 65 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

RX(1) OF 11 **A** + **B** ===> **C**



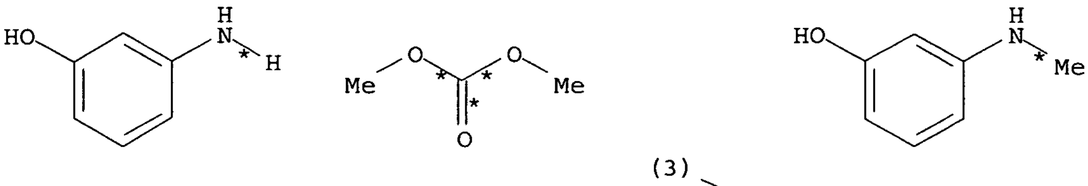
RX(1) RCT A **123-30-8**, B **616-38-6**
PRO C **150-75-4**
SOL 75-05-8 MeCN
CON 24 hours, 81 deg C
NTE chemoselective, optimization study, NaY zeolite used

RX(2) OF 11 **E** + **B** ===> **F**



RX(2) RCT E **95-55-6**, B **616-38-6**
PRO F **611-24-5**
SOL 616-38-6 Me2CO3
CON 3 hours, 90 deg C
NTE chemoselective, optimization study, NaY zeolite used

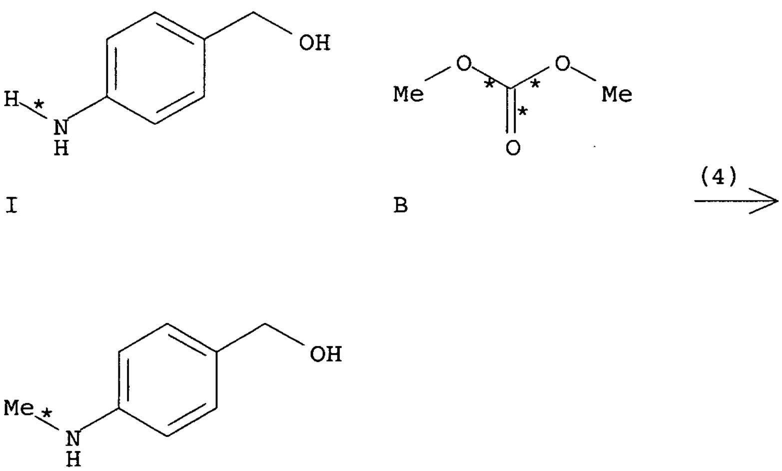
RX(3) OF 11 **G** + **B** ===> **H**



G B H
YIELD 96%

RX(3) RCT G **591-27-5**, B **616-38-6**
PRO H **14703-69-6**
SOL 616-38-6 Me2CO3
CON 3 hours, 90 deg C
NTE chemoselective, NaY zeolite used

RX(4) OF 11 **I** + **B** ===> **J**

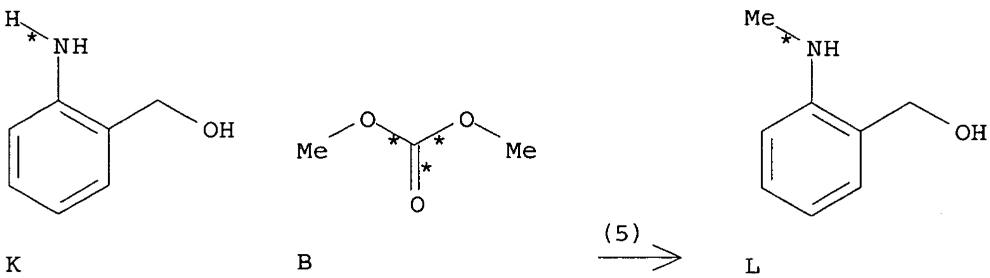


I B

J
YIELD 85%

RX(4) RCT I **623-04-1**, B **616-38-6**
PRO J **181819-75-0**
SOL 616-38-6 Me2CO3
CON 8 hours, 90 deg C
NTE chemoselective, NaY zeolite used

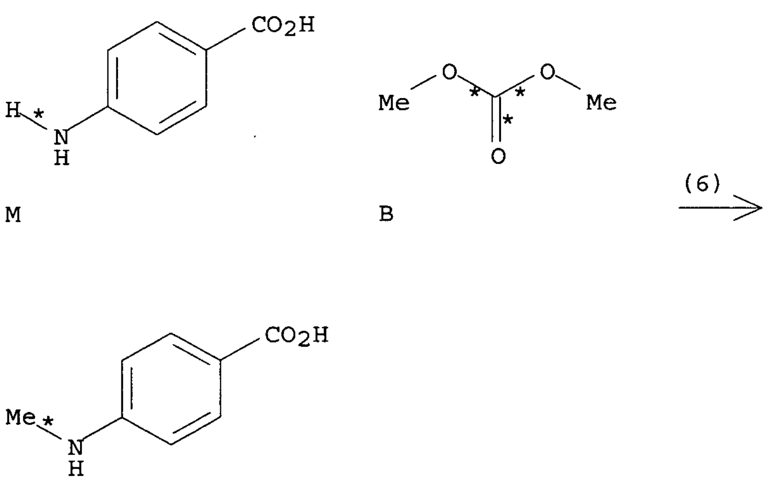
RX(5) OF 11 **K** + **B** ===> **L**



K B L
YIELD 98%

RX(5) RCT K **5344-90-1**, B **616-38-6**
PRO L **29055-08-1**
SOL 616-38-6 Me2CO3
CON 12 hours, 130 deg C
NTE chemoselective, NaY zeolite used

RX(6) OF 11 **M** + **B** ===> **N**

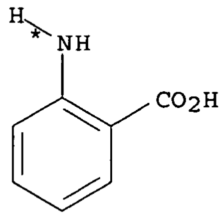


M B

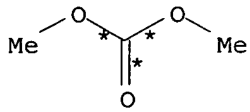
N
YIELD 90%

RX(6) RCT M **150-13-0**, B **616-38-6**
PRO N **10541-83-0**
SOL 616-38-6 Me2CO3
CON 9 hours, 90 deg C
NTE chemoselective, NaY zeolite used

RX(7) OF 11 **O** + **B** ===> **P**

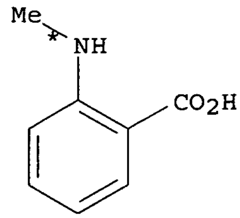


O



B

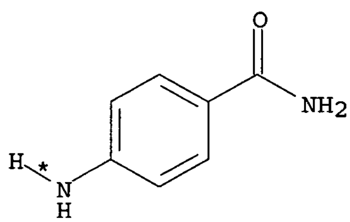
(7) →



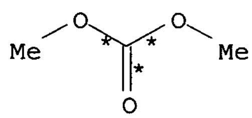
P
YIELD 90%

RX(7)	RCT	O **118-92-3**, B **616-38-6**
	PRO	P **119-68-6**
	SOL	616-38-6 Me2CO3
	CON	5 hours, 150 deg C
	NTE	chemoselective, NaY zeolite used

RX(8) OF 11 **Q** + **B** ===> **R**

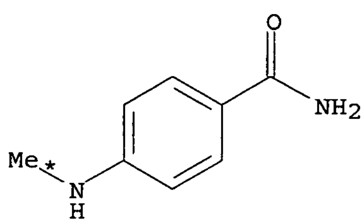


Q



B

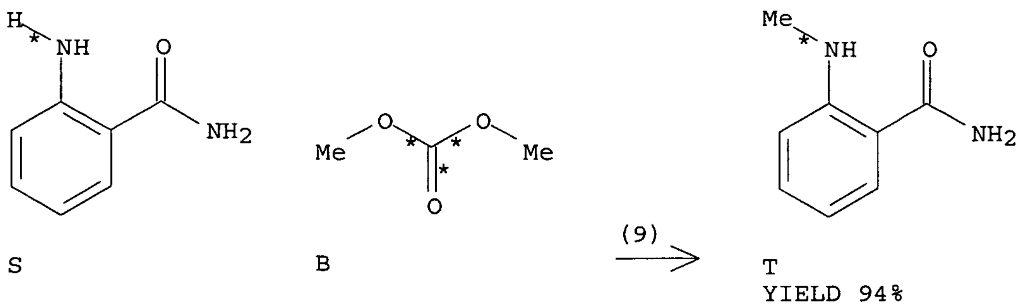
(8) →



R
YIELD 89%

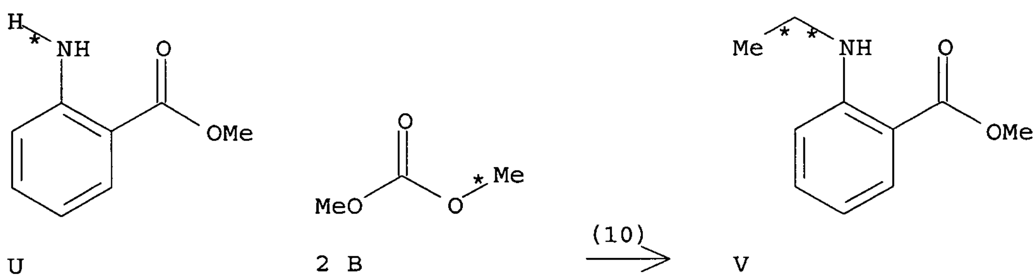
RX(8)	RCT	Q **2835-68-9**, B **616-38-6**
	PRO	R **38359-26-1**
	SOL	616-38-6 Me2CO3
	CON	24 hours, 90 deg C
	NTE	chemoselective, NaY zeolite used

RX(9) OF 11 **S** + **B** ===> **T**



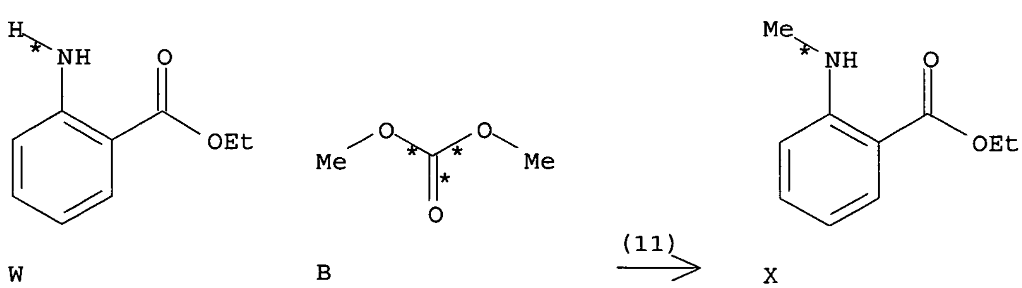

```
RX(9)      RCT  S 88-68-6, B 616-38-6
           PRO  T 7505-81-9
           SOL  616-38-6 Me2CO3
           CON  22 hours, 90 deg C
           NTE  chemoselective, NaY zeolite used
```

RX(10) OF 11 **U** + 2 **B** ===> **V**




```
RX(10)     RCT  U 134-20-3, B 616-38-6
           PRO  V 17318-49-9
           SOL  616-38-6 Me2CO3
           CON  5 hours, 150 deg C
           NTE  chemoselective, NaY zeolite used
```

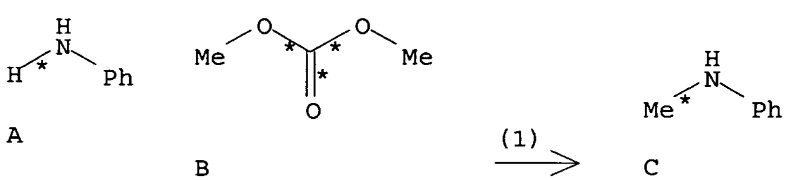
RX(11) OF 11 **W** + **B** ===> **X**



RX(11) RCT W **87-25-2**, B **616-38-6**
PRO X **35472-56-1**
SOL 616-38-6 Me2CO3
CON 5 hours, 150 deg C
NTE chemoselective, NaY zeolite used
AU **Selva, Maurizio**; **Tundo, Pietro**; Perosa, Alvise

L61 ANSWER 6 OF 9 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 111:25255 CASREACT
TITLE: Gas-liquid phase-transfer catalysis: a new continuous-flow method in organic synthesis
AUTHOR(S): **Tundo, Pietro**; Moraglio, Giovanni; Trotta, Francesco
CORPORATE SOURCE: Ist. Chim. Ind., Univ. Messina, Messina, 98010, Italy
SOURCE: Industrial & Engineering Chemistry Research (1989), 28(7), 881-90
CODEN: IECRED; ISSN: 0888-5885
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A new synthetic method, gas-liquid phase-transfer catalysis (GL-PTC), is described. GL-PTC is a continuous-flow procedure where gaseous reagents flow through a molten PT catalyst supported on a solid; no solvent is used. Reactions may develop through new mechanistic pathways compared with those of classical conditions; moreover, the reactions often occur in very selective ways. Some typical syntheses carried out under GL-PTC conditions are described in detail. They include syntheses of 2-alkylmalonic acid esters from the corresponding malonic acid esters, the reactions of Me2CO3, which give N-methylanilines from anilines and anisoles from phenols, and transesterification of EtOAc.

RX(1) OF 5 **A + B ===> C**

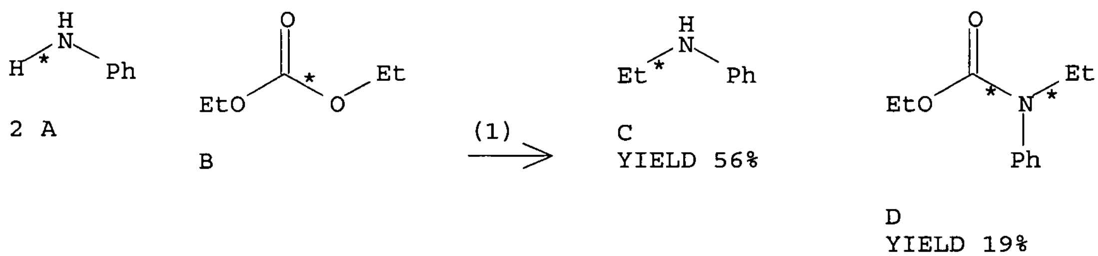


RX(1) RCT A **62-53-3**, B **616-38-6**
PRO C **100-61-8**
AU **Tundo, Pietro**; Moraglio, Giovanni; Trotta, Francesco

L61 ANSWER 7 OF 9 CASREACT COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 106:155960 CASREACT
TITLE: Selective mono-N-alkylation of aromatic amines by dialkyl carbonate under gas-liquid phase-transfer catalysis (GL-PTC) conditions
AUTHOR(S): Trotta, F.; **Tundo, P.**; Moraglio, G.
CORPORATE SOURCE: Ist. Chim. Org., Univ. Torino, Turin, 10125, Italy
SOURCE: Journal of Organic Chemistry (1987), 52(7), 1300-4
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The reaction between aromatic amines and dialkyl carbonates, carried out in

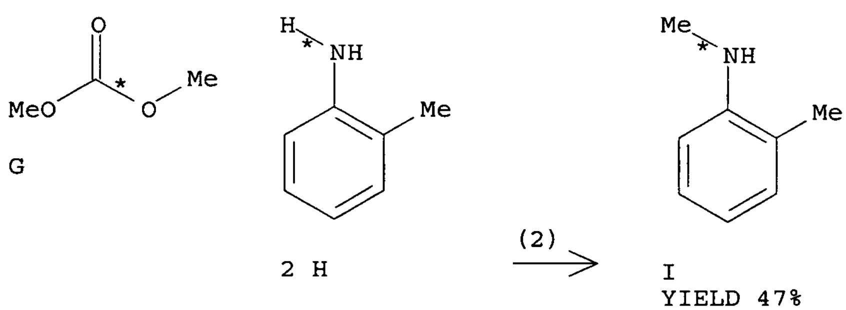
gas phase under GL-PTC conditions, in a continuous-flow process, produces the mono-N-alkylation product and its carboxyalkyl derivative with selectivity > 99%. The catalyst is a polyethylene glycol in the presence of K2CO3. The reaction produces CO2, which is removed from the system, so that the rwaction can be carried out indefinitely because the chemical nature of the catalytic bed, at steady state, does not change with time. The mechanism and selectivity were discussed.

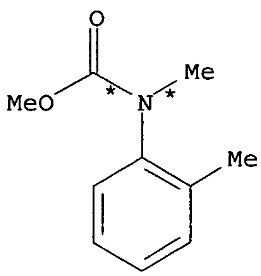
RX(1) OF 11 2 **A** + **B** ===> **C** + D



RX(1) RCT A **62-53-3**, B **105-58-8**
RGT E 584-08-7 K2CO3
PRO C **103-69-5**, D 1013-75-8
CAT 25322-68-3 HOCH2CH2OH polymer

RX(2) OF 11 **G** + 2 **H** ===> **I** + J

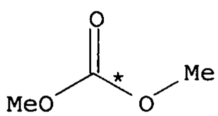




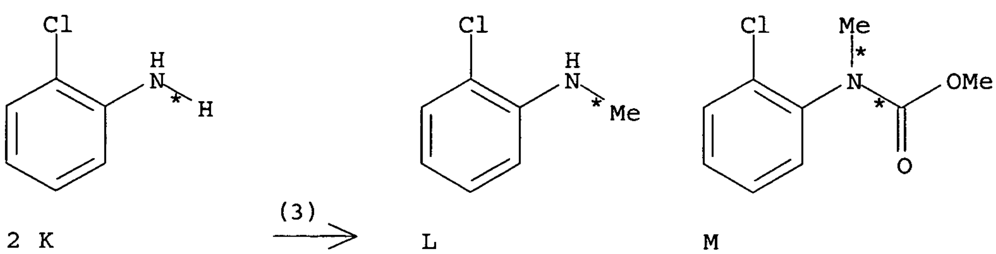
J
YIELD 25%

RX(2)   RCT G **616-38-6**, H **95-53-4**
         RGT E 584-08-7 K2CO3
         PRO I **611-21-2**, J 104189-18-6
         CAT 25322-68-3 HOCH2CH2OH polymer

RX(3) OF 11   **G** + 2 **K** ===> **L** + M



G



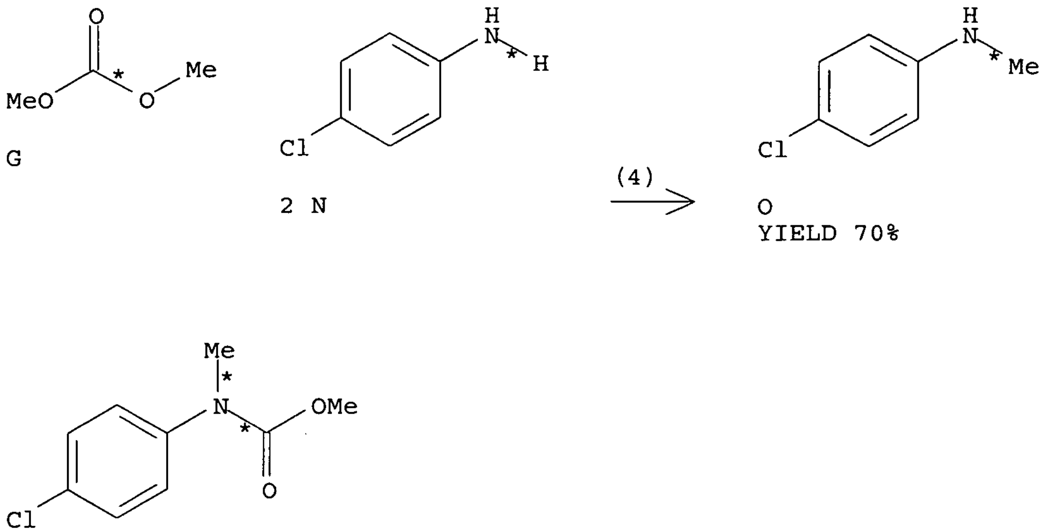
2 K

L
YIELD 62%

M
YIELD 22%

RX(3)   RCT G **616-38-6**, K **95-51-2**
         RGT E 584-08-7 K2CO3
         PRO L **932-32-1**, M 106712-21-4
         CAT 25322-68-3 HOCH2CH2OH polymer

RX(4) OF 11   **G** + 2 **N** ===> **O** + P



G

2 N

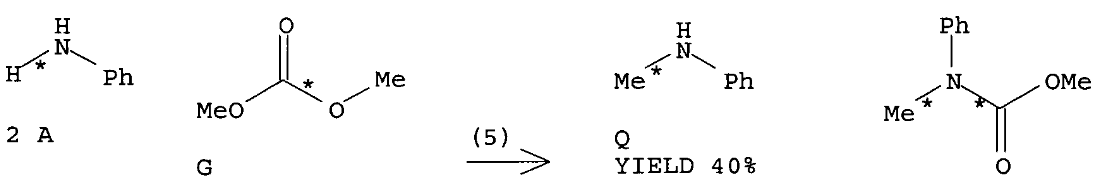
(4)

O
YIELD 70%

P
YIELD 19%

RX(4)   RCT G **616-38-6**, N **106-47-8**
           RGT E 584-08-7 K2CO3
           PRO O **932-96-7**, P 60561-39-9
           CAT 25322-68-3 HOCH2CH2OH polymer

RX(5) OF 11   2 **A** + **G** ===> **Q** + R



2 A

G

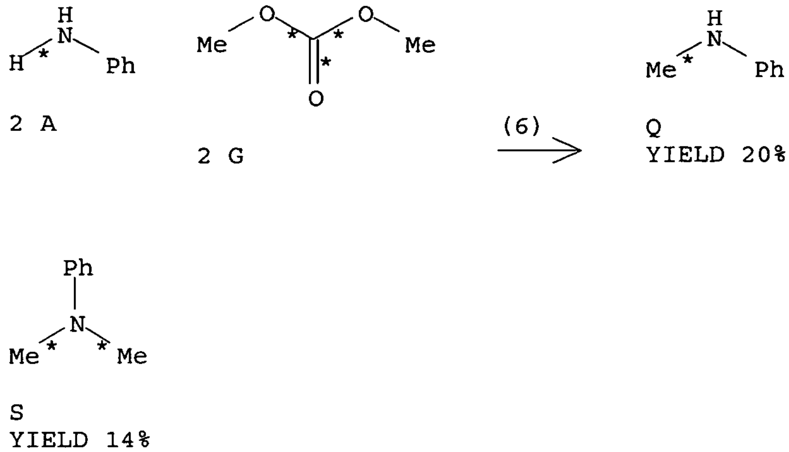
(5)

Q
YIELD 40%

R
YIELD 3%

RX(5)   RCT A **62-53-3**, G **616-38-6**
           RGT E 584-08-7 K2CO3
           PRO Q **100-61-8**, R 28685-60-1
           CAT 25322-68-3 HOCH2CH2OH polymer

RX(6) OF 11   2 **A** + 2 **G** ===> **Q** + S

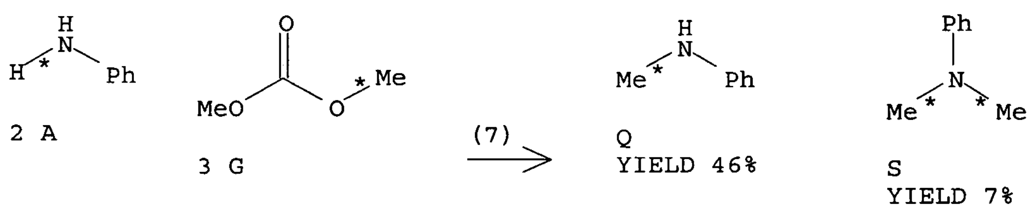


2 A 2 G (6) Q YIELD 20%

S YIELD 14%

```
RX(6)      RCT  A 62-53-3, G 616-38-6
           PRO  Q 100-61-8, S 121-69-7
           CAT  7631-86-9 SiO2
```

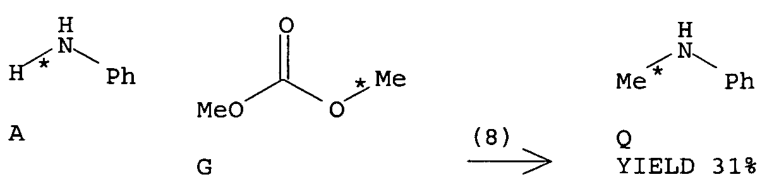
RX(7) OF 11   2 **A** + 3 **G** ===> **Q** + S



2 A 3 G (7) Q YIELD 46% S YIELD 7%

```
RX(7)      RCT  A 62-53-3, G 616-38-6
           PRO  Q 100-61-8, S 121-69-7
           CAT  1344-28-1 Al2O3
```

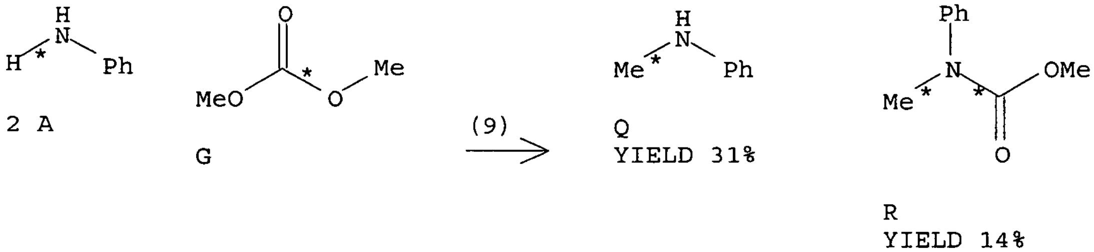
RX(8) OF 11   **A** + **G** ===> **Q**



A G (8) Q YIELD 31%

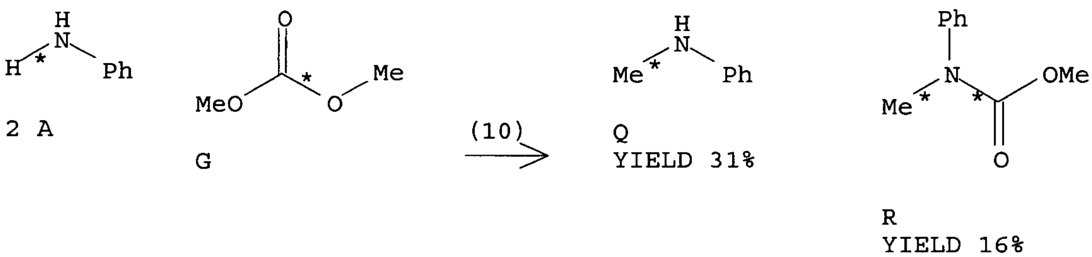
```
RX(8)      RCT  A 62-53-3, G 616-38-6
           RGT  U 1344-28-1 Al2O3
           PRO  Q 100-61-8
           CAT  3115-68-2 Bu4P.Br
```

RX(9) OF 11   2 **A** + **G** ===> **Q** + R



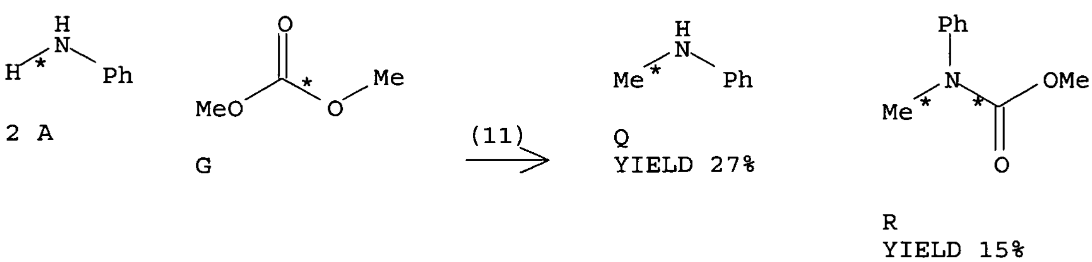
RX(9)   RCT A **62-53-3**, G **616-38-6**
          RGT E 584-08-7 K2CO3
          PRO Q **100-61-8**, R 28685-60-1
          CAT 25322-68-3 HOCH2CH2OH polymer
          SOL 110-82-7 Cyclohexane

RX(10) OF 11   2 **A** + **G** ===> **Q** + R



RX(10)   RCT A **62-53-3**, G **616-38-6**
          RGT E 584-08-7 K2CO3
          PRO Q **100-61-8**, R 28685-60-1
          CAT 25322-68-3 HOCH2CH2OH polymer
          SOL 109-99-9 THF

RX(11) OF 11   2 **A** + **G** ===> **Q** + R



RX(11)   RCT A **62-53-3**, G **616-38-6**
          RGT E 584-08-7 K2CO3
          PRO Q **100-61-8**, R 28685-60-1

CAT 25322-68-3 HOCH2CH2OH polymer
SOL 123-91-1 Dioxane
AU Trotta, F.; **Tundo, P.**; Moraglio, G.

L61 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2002:894442 CAPLUS
DOCUMENT NUMBER: 138:106304
TITLE: Mono-N-methylation of Primary Amines with Alkyl Methyl **Carbonates** over Y **Faujasites.** 2. Kinetics and Selectivity
AUTHOR(S): **Selva, Maurizio**; **Tundo, Pietro**; Perosa, Alvise
CORPORATE SOURCE: Dipartimento di Scienze Ambientali, Universita Ca' Foscari, Venice, 30123, Italy
SOURCE: Journal of Organic Chemistry (2002), 67(26), 9238-9247 CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB In the presence of a Na-exchanged Y faujasite, the reaction of primary aromatic amines 1 with 2-(2-methoxyethoxy)methylethyl **carbonate** [MeO(CH2)2O(CH2)2OCO2Me, 2a] yields the corresponding mono-N-Me derivs. ArNHMe with selectivity up to 95%, at substantially quant. conversions. At 130 °C, the reaction can be run under diffusion-free conditions and is strongly affected by the solvent polarity: for instance, in going from xylene (εr = 2.40) to triglyme (εr = 7.62) as the solvent, the pseudo-first-order rate constant for the aniline (1a) disappearance shows a 5-fold decrease. In DMF (εr = 38.25), the same reaction does not occur at all. Competitive adsorption of the solvent and the substrate onto the catalytic sites accounts for this result. The behavior of alkyl-substituted anilines ZC6H4NH2 [Z = p-Me, p-Et, p-Pr, p-(n-Bu) (1b-e); Z = 3,5-di-tert-butyl- and 2,4,6-tri-tert-butylanilines (1f,g)] and p-alkoxyanilines p-ZC6H4NH2 [Z = OMe, OEt, OPr, O-n-Bu (1b'-e')] clearly indicates a steric effect of ring substituents: as diffusion of the amine into the catalytic pores is hindered, the reaction hardly proceeds and the mono-N-Me selectivity (SM/D) drops as well. Moreover, the strength of adsorption of the amine onto the catalyst influences the rate and the selectivity as well: the reaction of p-anisidine and p-toluidine - despite the higher nucleophilicity of these compds. - is slower and even less selective with respect to aniline. From a mechanistic viewpoint, the intermediacy of carbamates ArN(Me)CO2R [R = MeO(CH2)2O(CH2)2] is suggested. At 90 °C, the reaction of benzylamine (7)-a model for aliphatic amines-with di-Me **carbonate** shows that the reaction outcome can be improved by tuning the amphoteric properties of the catalyst: in going from CsY to the more acidic LiY zeolite, methylation is not only more selective (SM/D ratio increases from 77% to 84%) but even much faster (CsY, conversion of 36% after 22 h; LiY, conversion of 43% after 7 h).

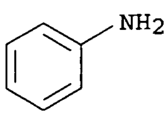
CC 22-4 (Physical Organic Chemistry)
ST amine methylation alkyl methyl **carbonate faujasite** catalyst kinetics selectivity
IT Y zeolites
RL: CAT (Catalyst use); USES (Uses)
(CsY; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT **Faujasite**-type zeolites
RL: CAT (Catalyst use); USES (Uses)

(LiY; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT Y zeolites
RL: CAT (Catalyst use); USES (Uses)
(Na-exchanged; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT **Amines, reactions**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(**aromatic**; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT Methylation catalysts
Methylation kinetics
Solvent polarity effect
Substituent effects
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT Zeolite KY
RL: CAT (Catalyst use); USES (Uses)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT Dielectric constant
(of methoxyethoxymethylethyl **carbonate**; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT Methylation
(regioselective, of amines; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT **62-53-3**, Aniline, reactions 100-46-9, Benzylamine, reactions 104-13-2, p-Butylaniline 104-94-9, p-Anisidine 106-49-0, p-Toluidine, reactions 156-43-4, p-Phenetidine 589-16-2, p-Ethylaniline 961-38-6, 2,4,6-Tri-tert-butylaniline 2380-36-1, 3,5-Di-tert-butylaniline 2696-84-6, p-Propylaniline 4344-55-2, 4-Butoxyaniline 4469-80-1, 4-Propoxyaniline 141814-27-9
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT 99-97-8 100-61-8, N-Methylaniline, formation (nonpreparative) 103-67-3 103-83-3 623-08-5 701-56-4 4150-37-2 5817-70-9 5961-59-1 37846-06-3 56475-82-2
RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT 1864-93-3 2909-79-7 3154-18-5 5279-59-4 13330-29-5 23563-27-1 36373-76-9 71089-15-1 137273-36-0 485795-30-0 485795-31-1 485795-32-2 485799-00-6
RL: FMU (Formation, unclassified); PRP (Properties); FORM (Formation, nonpreparative)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT 616-38-6, Dimethyl **carbonate**
RL: RCT (Reactant); RACT (Reactant or reagent)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)
IT 68-12-2, DMF, uses 112-49-2, Triglyme 1330-20-7, Xylene, uses
RL: NUU (Other use, unclassified); USES (Uses)

(solvent; kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)

IT **62-53-3**, Aniline, reactions
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(kinetic study on mono-N-methylation of primary amines with alkyl Me **carbonates** over Y **faujasites**)

RN 62-53-3 CAPLUS
CN Benzenamine (9CI) (CA INDEX NAME)



REFERENCE COUNT: 41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

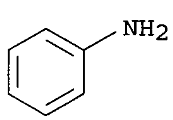
L61 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:271593 CAPLUS
DOCUMENT NUMBER: 127:17455
TITLE: Selective mono-N-methylation of primary aromatic amines by dimethyl **carbonate** over **faujasite** X- and Y-type zeolites
AUTHOR(S): **Selva, Maurizio**; Bomben, Andrea; **Tundo, Pietro**
CORPORATE SOURCE: Dip. Scienze Ambientali, Univ. Venezia, Venice, 2137-30123, Italy
SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1997), (7), 1041-1045
CODEN: JCPRB4; ISSN: 0300-922X
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English

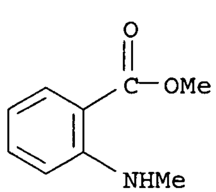
AB The reaction of di-Me **carbonate** (DMC) with different primary aromatic amines ArNH2 (Ar = Ph, 4-O2NC6H4, 4-NCC6H4, 2-MeO2CC6H4, 2,6-Me2C6H3) has been investigated under batch conditions (autoclave) in the presence of Y- and X-type zeolites. Operating at 120-150°C, highly selective mono-N-methylations are observed for anilines even when they are deactivated by either electronic effects or steric hindrance; typical selectivities for the formation of the corresponding mono-N-Me derivs. [ArNH(CH3)] are in the range 92-98%, at a substrate conversion of 72-93%. A synergic effect between the reactivity of DMC (acting both as a methylating and as a reversible methoxycarbonylating agent) and the dual acid-base properties of zeolites is considered to be responsible for the unusually high selectivity observed; accordingly, a reaction mechanism is discussed, involving carbamates (ArNHCO2CH3) and N-methylcarbamates [ArN(CH3)CO2CH3] as intermediates. The reaction is an example of a synthesis with low environmental impact: it couples the use of a non-toxic methylating agent (DMC, in place of the highly toxic Me halides or di-Me sulfate) with eco-friendly catalysts (zeolites) in a waste-free process.

CC 25-4 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)
ST amine arom methylation dimethyl **carbonate** zeolite
IT Methylation
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)

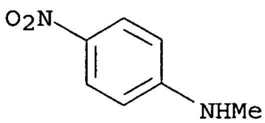
IT **Faujasite**-type zeolites
RL: CAT (Catalyst use); USES (Uses)
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)
IT **Amines, reactions**
RL: RCT (Reactant); RACT (Reactant or reagent)
(**aromatic**; N-methylation of **aromatic** amines by di-Me **carbonate** over **faujasite** zeolites)
IT **62-53-3**, Benzenamine, reactions 87-62-7, 2,6-Dimethylaniline 100-01-6, reactions 616-38-6, Dimethyl **carbonate** 619-45-4, Methyl 4-aminobenzoate 873-74-5, 4-Aminobenzonitrile
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)
IT **85-91-6P 100-15-2P 100-23-2P 100-61-8P**
, preparation **121-69-7P**, preparation **767-71-5P**
769-06-2P 1197-19-9P 1943-87-9P
2603-10-3P 4714-62-9P 7143-42-2P
10072-05-6P 20642-93-7P 28685-60-1P
94563-11-8P
RL: SPN (Synthetic preparation); **PREP (Preparation)**
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)
IT **62-53-3**, Benzenamine, reactions
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)
RN 62-53-3 CAPLUS
CN Benzenamine (9CI) (CA INDEX NAME)



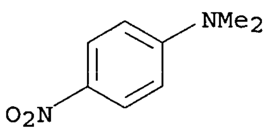
IT **85-91-6P 100-15-2P 100-23-2P 100-61-8P**
, preparation **121-69-7P**, preparation **767-71-5P**
769-06-2P 1197-19-9P 1943-87-9P
2603-10-3P 4714-62-9P 7143-42-2P
10072-05-6P 20642-93-7P 28685-60-1P
94563-11-8P
RL: SPN (Synthetic preparation); **PREP (Preparation)**
(N-methylation of aromatic amines by di-Me **carbonate** over **faujasite** zeolites)
RN 85-91-6 CAPLUS
CN Benzoic acid, 2-(methylamino)-, methyl ester (9CI) (CA INDEX NAME)



RN 100-15-2 CAPLUS
CN Benzenamine, N-methyl-4-nitro- (9CI) (CA INDEX NAME)



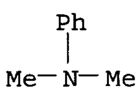
RN 100-23-2 CAPLUS
CN Benzenamine, N,N-dimethyl-4-nitro- (9CI) (CA INDEX NAME)



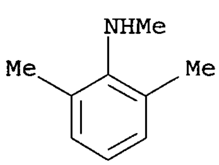
RN 100-61-8 CAPLUS
CN Benzenamine, N-methyl- (9CI) (CA INDEX NAME)

Me—NH—Ph

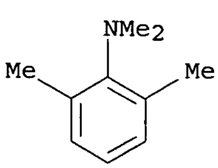
RN 121-69-7 CAPLUS
CN Benzenamine, N,N-dimethyl- (9CI) (CA INDEX NAME)



RN 767-71-5 CAPLUS
CN Benzenamine, N,2,6-trimethyl- (9CI) (CA INDEX NAME)

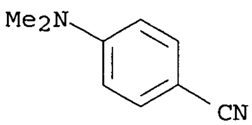


RN 769-06-2 CAPLUS
CN Benzenamine, N,N,2,6-tetramethyl- (9CI) (CA INDEX NAME)

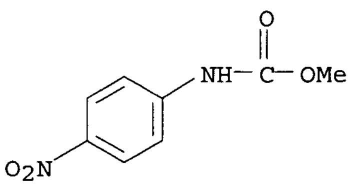


RN 1197-19-9 CAPLUS

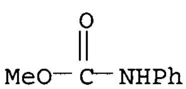
CN Benzonitrile, 4-(dimethylamino)- (9CI) (CA INDEX NAME)



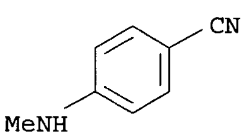
RN 1943-87-9 CAPLUS
CN Carbamic acid, (4-nitrophenyl)-, methyl ester (9CI) (CA INDEX NAME)



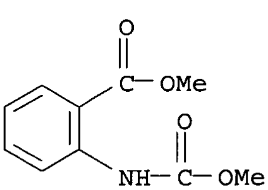
RN 2603-10-3 CAPLUS
CN Carbamic acid, phenyl-, methyl ester (9CI) (CA INDEX NAME)



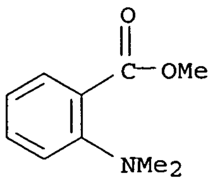
RN 4714-62-9 CAPLUS
CN Benzonitrile, 4-(methylamino)- (9CI) (CA INDEX NAME)



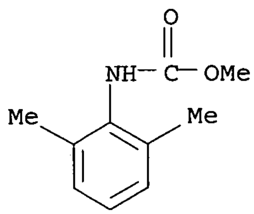
RN 7143-42-2 CAPLUS
CN Benzoic acid, 2-[(methoxycarbonyl)amino]-, methyl ester (9CI) (CA INDEX NAME)



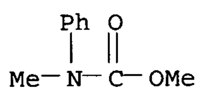
RN 10072-05-6 CAPLUS
CN Benzoic acid, 2-(dimethylamino)-, methyl ester (9CI) (CA INDEX NAME)



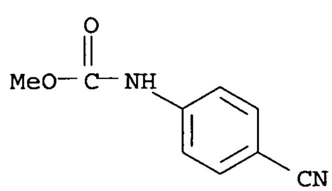
RN 20642-93-7 CAPLUS
CN Carbamic acid, (2,6-dimethylphenyl)-, methyl ester (9CI) (CA INDEX NAME)



RN 28685-60-1 CAPLUS
CN Carbamic acid, methylphenyl-, methyl ester (9CI) (CA INDEX NAME)



RN 94563-11-8 CAPLUS
CN Carbamic acid, (4-cyanophenyl)-, methyl ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=>